Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

PROCEEDINGS - CANMET/EEC MEETING ON PROCESSING OF COMPLEX MINERALS
MAY 13-14, 1985

W.A. Gow
Mineral Sciences Laboratories

June 1985

Projects: MRP-3.0.1.3.04
Improved Recovery Technology of Fine Particles

MRP-3.0.1.3.05
Liberation/Flotation Models

MRP-3.0.7.7.01
Silver Recovery in the Zinc Industry

111pp

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT MRP/MSL 85-94(TR)

This document is an unedited interim report prepared for limited circulation and review within the author's branch and for the opinion of the Canada Centre for Mineral and Energy Technology (CANMET)

Ce document est un rapport provisoire non-révisé et rédigé principalement pour fin de circulation et de commentaire à l'intérieur du service auteur et pour opinion de la Centre canadien de la technologie des minéraux et de l'énergie (CANMET)

MICROMEDIA

OCT 11 1985

TABLE OF CONTENTS

	Page
Preface	ii
Agreed joint minutes	1
Agenda	4
Attendees	6
Annex I: Session Summaries	8
Session I: Liberation Modelling/Flotation	
a) Canmet (Petruk)	9
b) Warren Spring: Flotation (Cutting)	12
Simulation	17
c) Université Libre de Bruxelles (Panou)	21
d) BRGM (Broussaud)	43
e) Imperial College (Jones)	49
Session II: Fine Particle Technology	
a) Canmet (Sirois)	53
b) Minemet Recherche (Predali)	56
c) Warren Spring (Parsonage)	58
d) Université de Nancy (Cases)	62
Session III: Iron Control and Elimination in Hydrometallurgy	
i) Canmet (Dutrizac)	65
ii) Imperial College (Monhemius)	76
iii) Minemet (Ricalens)	78
iv) L'Air Liquide (Burzynski)	80
Annex II: Technical Research Proposals (EEC/CANMET)	
Liberation Process Modelling	92
Fine Particle Technology	99
Iron Control in Hydrometallurgy	103

PREFACE

Joint interest on the part of Canmet and the EEC in cooperative research in the treatment of complex minerals dates back to 1981. In October 1982, Canmet hosted a Canada/European Economic Community Seminar on the Treatment of Complex Minerals, the proceedings of which were made available as Canmet report MRP/MSL 83-101(OP). At that seminar it was realized that there were many common problems and R&D interest. Therefore discussions began to develop mechanisms for cooperative R&D in this field. Three areas of common interest were identified as:

i) Liberation modelling/flotation
ii) Fine particle technology
iii) Iron control and elimination in hydrometallurgy.

In May 1984, four Canmet scientists and a representative from a Canadian mining company attended the meeting in Brussels at which EEC contractors presented progress reports on research in thse three areas.

As a result of these meetings it was agreed that there was a basis for developing a cooperative Canmet/EEC research program. Therefore, the EEC agreed to hold their 1985 presentations on contract progress in Canada and to use the occasion to develop more comprehensive joint objectives. This report is a summary of the meeting held in Ottawa May 13-14, 1985. Over the balance of 1985 both Canmet and the EEC will more fully develop specific research plans related to these objectives and reach an agreed integrated program of research.

This report provides the agreed joint minutes, summaries of technical presentations in Annex I and technical research proposals in Annex II. Readers are encouraged to comment as they see fit.

W.A. Gow,
Director,
Mineral Sciences Laboratories.

Agreed Joint Minutes

Canada/EEC Meeting on Processing of Complex Minerals

May 13-14, 1985

Camsell Hall, 588 Booth Street, Ottawa

The meeting was opened by Mr. W.A. Gow, Director of Mineral Sciences Laboratories, CANMET. In his presentation, Mr. Gow recalled the work leading up to the organization of this joint meeting. The first report was recorded in October 1982 when CANMET hosted a Canada/EEC seminar in Ottawa on the processing of complex minerals. The Canadian mineral industry was well represented at the meeting and contributed significantly to the proceedings.

As a result of that seminar, both parties (CANMET/EEC) expressed interest in developing a mechanism for conducting a Canada/EEC cooperative R&D program, and the EEC invited CANMET staff to a workshop in Brussels on complex minerals in May 1984. In addition, Mr. Gow stressed the willingness and interest of CANMET to cooperate with the EEC in the field of non fuel raw materials.

The Commission's representative (Mr. Donato) in its presentation recalled the background that led the EEC to launch in 1978 the first R&D program followed by a second one in 1982 which is going to be terminated at the end of this year. It has also presented some ideas that so far have been included in working technical documents, of the Commission which cover the Liberation/modelling/ Flotation; Fine particle technology and iron elimination and control in hydrometallurgy. It was also announced that a possible four -year R&D program on raw materials could start in 1986.

Session I: Liberation modelling/flotation
Under this point reports were presented by:

Dr. W. Petruk, CANMET
Mr. G. Cutting, Warren Spring Laboratory
Prof. G. Panou, Universite Libre de Bruxelles
M. A. Broussaud, BRGM (Orleans, France)
Prof. M.P. Jones, Imperial College, London, U.K.

This session was concluded with a discussion in which many speakers expressed an interest in the work performed by EEC contractors and CANMET researchers (in Annex I are technical papers presented by the various speakers).

Session II: Fine particle technology

Under this point of the agenda various papers were presented by:

Mr. L.L. Sirois, CANMET
Mr. J.J. Predali, Minimet Recherche, France
Mr. P. Parsonage, Warren Spring Laboratory, U.K.
Prof. J.M. Cases, Polytechnique de Lorraine, Nancy, France

(read by Mr. Predali)

The related papers to this session are also annexed to this report. (Annex I)

Before ending this session a discussion was developed in which Canadian and EEC contributors exchanged ideas and expressed interest for future joint cooperation.

Session III: Iron control and elimination in hydrometallurgy

Under this point reports were presented by:

Dr. J.E. Dutrizac, CANMET
Dr. A.J. Monhemius, Imperial College, London, U.K.
Mr. J.P. Burzynski, L'Air Liquide, France
Mr. M. Richalens, Minimet Recherche, France

A concluding discussion on this topic showed that past research activity developed by CANMET and EEC contractors has many various common points and it was said that joint cooperation in the field could lead to processes having reduced costs for iron control, elimination and possibly recovery for other industrial uses. Papers presented in Session II are in Annex I.

Discussion of Research Objectives

Session IV: Priorities, Schedules and Performers

Under this point the Commission representative, after consultations with CANMET representatives, suggested the attendees form three working parties to develop, if possible, specific joint or parallel research projects for the three major areas under discussion, namely:

- Liberation/Modelling
- Fine Particles Technology
- IRON Control in Hydrometallurgy

In addition to the above lines of research, a new suggestion was made concerning linear analysis with microprobe image analysis. This point was presented by Prof. Jones from Imperial College and received support from CANMET.

As a practical approach it was agreed that the three working parties would identify possible research projects which would be partly covered by future contractors to the EEC and partly covered by CANMET and other Canadian research groups.

Projects could have a maximum duration time of 36 months. Annual joint meetings should be organized alternately in Ottawa and in Brussels. At the end of the project a final joint meeting will analyse the results in order to exchange reports and information and suggest further cooperative research projects. When the 1986-1989 (EEC) program on non fuel minerals is developed, the various organizations present at this (May 14, 1985) meeting (and possibly others) will present proposals to be submitted to the Commission which as usual will consult the related CGC in order to collect opinions and comments. Only after this procedure is completed could scientific confirmation be agreed to with CANMET.

When the Council of EEC Ministers has adopted the research programme proposed by the Commission, CANMET will take the initiative by an exchange of letters with the Commission or other means to formalize a cooperative arrangement between CANMET and the EEC.

In conclusion, the best concept for developing cooperation is that each side will put up its own funds to work toward a common specific objective. In the EEC, the work will be performed under contract to research organizations, in a number of countries, while in Canada the work will be done in house by CANMET or other organizations under contract to CANMET.

Attached please find the technical research proposals developed by the three working parties on Liberation/Modelling, Fine Particles Technology, Iron Control and Elimination in Hydrometallurgy and applications of linear analysis with microprobe image analyses.

Annex I - Papers presented in the three sessions.

Annex II - Technical Research Proposals (EEC/CANMET)

W. A. Gow
Director, MSL
CANMET.

AGENDA

Canada/EEC Meeting on Processing of Complex Minerals

May 13-14, 1985

Camsell Hall
588 Booth Street

May 13, 1985

09:00h Registration

09:30h Welcome: Mr. W.A. Gow
Director
Mineral Sciences Laboratories
Canada Centre for Mineral and Energy Technology (CANMET)
Department of Energy, Mines and Resources

Introduction: Mr. M. Donato
Head
Specialized Services
Raw Materials Division
Directorate G XII
European Economic Commission

09:50h Session I: Liberation Modelling
Introduction: W. Petruk, CANMET
Reports: G. Cutting, Warren Spring Laboratory
G. Panou, Universite Libre de Bruxelles
A. Broussaud, BRGM
M.P. Jones, Imperial College

10:15-10:30h Coffee Break

12:00-14:00h Lunch - Ad Lib
(Speak to CANMET people about local restaurants)

14:00h Session II: Fine Particle Technology
Introduction: L.L. Sirois, CANMET
Reports: J.J. Predali, Minemet Recherche
F. Richalens, Minemet Recherche
P. Parsonage, Warren Spring Laboratory
J.M. Cases, Polytechnique de Lorraine-Nancy

15:00-15:30h Coffee Break

15:30h Session III: Iron Control in Hydrometallurgy
Introduction: J.E. Dutrizac, CANMET
Reports: A.J. Monhemius, Imperial College
J. Burzynski, L'Air Liquide

18:30h Reception and Dinner (19:15h)
Hosted by CANMET
Skyline Hotel (Top Floor)

May 14, 1985

09:00h Session IV: Discussion of Research Objectives, Priorities, Schedules and Performers
Introduction: Mr. M. Donato

Note: Mr. Donato will organize the discussion of each of the three topics in the previous sessions.

10:15-10:30h Coffee Break

12:00h Lunch - Ad Lib

13:30h Session V: Closing Discussion and Concluding Remarks

15:00h Adjournment

ATTENDEES AT CANADA/EEC MEETING
MAY 13-14, 1985

EUROPEAN DELEGATES

Broussaud, A.	BRGM
Burzynski, J.	L'Air Liquide
Cutting, G.W.	Warren Spring
Donato, M.	EEC
Jones, M.P.	Imperial College
Monhemius, A.J.	Imperial College
Panou, G.	Université Libre
Parsonage, P.	Warren Spring
Predali, J.J.	MINEMET Recherche
Ricalens, F.	MINEMET Recherche

CANADIAN DELEGATES

Adorjan, A.	Technical University of Nova Scotia
Barbery, G.	Laval University
Barlin, B.	Hudson Bay Mining & Smelting
Beattie, M.	Bacon, Donaldson & Associates
Campbell, M.C.	CANMET, Energy, Mines & Resources Canada
Craigen, W.J.S.	CANMET, Energy, Mines & Resources Canada
Cristovici, M.	CANMET, Energy, Mines & Resources Canada
Dutrizac, J.E.	CANMET, Energy, Mines & Resources Canada
Finch, J.	McGill University
Gow, W.A.	CANMET, Energy, Mines & Resources Canada
Greer, R.	Kidd Creek
Hendricks, D.	Brunswick Mining & Smelting
Laguitton, D.	CANMET, Energy, Mines & Resources Canada
Lakshmanan, V.I.	Ontario Research Foundation
MacDonald, R.J.C.	CANMET, Energy, Mines & Resources Canada
McKay, D.R.	Cominco
Madge, D.	Kilborn (Saskatchewan) Limited
Mathieu, G.	CANMET, Energy, Mines & Resources Canada
Mouland, J.	Kidd Creek
Mular, A.	University of British Columbia
Petruk, W.	CANMET, Energy, Mines & Resources Canada
Poling, G.	University of British Columbia

Sabourin, R.G.	CANMET, Energy, Mines & Resources Canada
Scott, D.	Kidd Creek
Sirois, L.L.	CANMET, Energy, Mines & Resources Canada
Smith, H.W.	University of Toronto
Spira, P.	Noranda Research
Stefanski, M.	CANMET, Energy, Mines & Resources Canada
Stemerowicz, A.	CANMET, Energy, Mines & Resources Canada
Stowe, K.	Noranda Research
Therien, M.	Energy & Resources (Quebec)
Wyslouzil, B.	Lakefield Research

ANNEX I

SESSION I. Mineral Liberation Modelling

Papers presented by :

a)	CANMET	: W. PETRUCK
b)	W.S.L.	: G.W.CUTTING (2)
c)	Univ. Libre Bruxelles	: G. PANOU
d)	B.R.G.M.	: A. BROUSSAUD
e)	Imperial College	: M.P. JONES

SESSION II. Fine Particle Technology

Papers presented by :

a)	CANMET	: D. L. SIROIS
b)	Minemet recherche +	: J.J. PREDALI
	Univ. de Nancy	: G. CASES
c)	W.S.L.	: P. PARSONAGE

SESSION III. Iron Control in Hydrometallurgy

Papers presented by :

a)	CANMET	: J.E. DUTRIAZC
b)	Imperial College	: A.J. MONHEMIUS
c)	Minemet Recherche	: M. RICALENS
d)	L'Air Liquide	: J. BURZIINSKI

MODELLING ON LIBERATION/FLOTATION OF ORES IN INDUSTRIAL PLANTS

by

WILLIAM PETRUK
CANMET, 555 BOOTH ST., OTTAWA, ONT. CANADA

INTRODUCTION

I was asked to present a general outline of the program "modelling on liberation/flotation of ores in industrial plants", and to give a progress report on current work at CANMET and Canada on this topic. A good outline of the program was given at the 1984 EEC meeting in Brussels by Mr. A. Broussaud of BRGM. He stated that at BRGM the activities covered by the topic are:

1. Mineral balancing,
2. Mineral liberation,
3. Development of models for flotation and liberation, and
4. Comparison of calculated or laboratory results with industrial results.

I interpret that these steps cover the spectrum of the program under discussion, and they are in accord with a project that has been approved for study at CANMET.

Mineral Balancing

Mr. Broussaud of BRGM pointed out that both BRGM and Warren Springs Laboratory had independently developed computer programs to establish self consistent material balances of industrial plants from inconsistent and overabundant data. Both programs have the same objectives but not the same methodologies.

At CANMET we have a project for developing and refining a materials balance program as part of our SPOC (simulated procedures for ore and coal) project. We have developed and refined a general materials balance program (MATBAL 3) and have developed a sequential materials balance program (BILMAT). MATBAL 3 is univeral program that can balance simple to complex circuits by using either a few or many readings. It can be used to balance grinding circuits, flotation circuits and a plant.

BILMAT is designed to perform a materials balance in sequence. For example, if we wanted to perform a materials balance for free chalcopyrite and free bornite we must first perform a materials balance for the total wt % of material at each point, secondly we must perform a materials balance for the quantity of copper at each point (chemical assay), thirdly balance the quantities of copper due to total chalcopyrite and total bornite, and finally balance the quantities of copper due to free chalcopyrite, to unliberated chalcopyrite, to free bornite and to unliberated bornite at each point. This sequence is necessary because we assay each sample for total copper, analyse it for total chalcopyrite and bornite, and then analyse it for free and unliberated mineral grains. The accuracy of the results decreases from the first to the last analysis. Therefore

each analysis must be balanced in the sequence given above.

Mineral Liberations

Since concentrators are designed to recover free minerals, it is important in liberation modelling to (1) develop methods for predicting liberations that would be obtained at certain grinds, (2) develop methods for accurately measuring the liberations in the ground ore, an (3) develop methods of assesssing the affect of free and unliberated grains on flotation. Methods that are in use for predicting liberations involve performing a linear size analysis of the minerals of interest in uncrushed specimens of the ore, and calculating the percentages of minerals that are free in each screened fraction of the ground ore. In many cases, however, due to different breakages of the minerals in different ore types, there is a poor correlation between the calculated liberations and measured values for screened fractions because of limitations on the models. At CANMET we have a program of collecting data sets which consist of linear size analysis for a variety of minerals and measuring mineral liberation in each screened fraction for a variety of ore types. This will establish a data base for either modifying the existing models or for developing new ones.

To determine the actual liberations that are obtained during grinding each screened fraction of the ground ore must be analysed with an image analyser. The 1984 EEC presentations at Brussels indicated that at Warren Springs Laboratory they perform this type of analysis by point counting. Image analysis, however, is much more accurate, more relaiable , and less tedious in performing the analysis. Dr. Jones performs linear analysis with a microprobe image analyser, whereas at BRGM and CANMET we do it by arial analysis with an image analyser interfaced with a scanning electron microscope. Mr. Broussaud reported that at BRGM a computer program was written to test the reliability of linear vs arial analysis in determining mineral liberations. The results showed that arial analysis gave truer data when analysing mill products. The author has had preliminary discussions with Dr. Jones aimed at carrying out a joint study that would evaluate this finding.

Development of Models for Flotation and Liberation

The presentations at the 1984 EEC meeting in Brussels showed that Warren Springs Laboratory and BRGM have independently developed computer simulation programs for predicting flotation plant performance. Developing a model requires a large amount of metallurgical data such as flotation reagents, flow, retention time, etc, and and data on mineral liberations. Generally the metallurgical data can be controlled and built into a computer program. The mineral liberations are generally assumed because too little is known about them. The strategy at CANMET is to incorporate the known metallurgical data, and to determine mineral liberations which includes the recovery of various sized free mineral grains, and the behaviour of the various types of

unliberated grains. We are in the process of collecting liberation data on various types of ores. When we collect enough data we will evaluate parameters that should be measured for flotation plant simulatuion.

Comparison of Calculated or Laboratory results with industrial plants

Liberation studies at CANMET are often performed on material from industrial plants and we generally determine the flow and recoveries of free mineral particles and unliberated mineral grains. These data provide a basis for modifying the plants and provide information for refining the liberation and flotation models.

(1)

DEPARTMENT OF TRADE AND INDUSTRY

Warren Spring Laboratory, Mineral Processing Division

March 1985: Project MSM 086; Modelling of Flotation Processes

Introduction and Background

The object of this project is the development and verification of mathematical models of continuously operated, multi-stage flotation process for flowsheet design purposes. Prior to the establishment of this project, WSL had developed a flotation process modelling technique, which was based upon a decoupling of the factors controlling activity in flotation cells into slurry phase and froth phase mechanisms. Great difficulty had been encountered in the development of flotation models of continuously operated plant generally due to the lack of good and reliable techniques to establish the parameters needed by the models from simple and effective laboratory-scale testwork on representative ore samples. In general, flotation processes are designed using the small quantity of laboratory-scale testwork results available coupled with generous helpings of professional judgement and expertise. Earlier attempts to improve the scope and quality of laboratory-scale data had not proved to be very successful and it was clear that modelling methods of immediate application to industrial problems must both rely largely upon and be capable of using, data derived from general expertise. In this light the flotation model should be viewed as a means whereby available laboratory-scale data is coupled with expertise and the model permits the design engineer to manipulate relevant available data to obtain estimations of likely process performance over extensive process flowsheet structures.

Most froth flotation engineers consider flotation response in terms of the activity occurring in the pulp or slurry phase and the effects of froth mechanisms. The model structure developed at WSL was based upon the use of first order rate functions in conjunction with stage residence times to describe the pulp phase mechanisms. Froth mechanisms (principally froth drainage effects) were introduced into the model in the form of stage efficiency factors. These models proved successful, both in terms of their ability to adequately model mass transport in multi-stage flotation processes and the utilisation of experience and judgement to supplement laboratory-scale testwork results.

Improvements in model utility were expected to come from improved understanding of the relevance of flotation rate functions, as derived from laboratory-scale testwork, and the influences of mechanisms operating in the froth phases upon the responses of flotation stages.

Progress

A large amount of data has been collected from operating flotation processes, both in terms of the mass transport of mineral species through the process flowsheet as produced by the flotation response in the cells and the mechanisms operating in the froth phases of selected cells in the processes. Feed material from the processing plants have been used to implement laboratory and pilot-scale testwork at WSL, where both flotation rate responses and froth drainage mechanisms have been investigated. This work has formed the subject of a number of technical publications. The principle findings and results from the above work are given below:

(1) Great difficulty has been encountered in directly relating the results obtained from laboratory-scale, flotation rate testwork on representative ore samples to the operational response of continuously operated flotation plant. Small differences in rate response are observed at laboratory-scale with respect to different mineral species (or particle size ranges) but these differences are insufficient to characterise the different responses observed for the mineral species on process plants using modelling methods based mainly upon flotation rate factors. Typically the froth discharges observed on the roughing bank of a complex sulphide mineral flotation plant, where the early cells in the bank are pyrite rich, the middle cells are sphalerite rich and the final cells are arsenopyrite rich.

(2) The results obtained from laboratory-scale testwork for a wide range of ore types (both sulphide and oxide types) all exhibit very similar rate coefficients. However, considerable differences occur in the ratio of the flotable fractions to the non-floating fractions of potentially floatable mineral species. These results indicate if a mineral species has been conditioned to float in a manner likely to be commercially exploitable then the flotation rate of the floatable fraction, as measured at laboratory scale, will lie within a narrow and prescribed range.

(3) Work implemented at pilot-scale, both in batch and continuously operated modes, has shown that the fraction floatable ratios dominate the flotation rate factors, the fraction floatable ratios change considerably under changed operational conditions either with or without further addition of reagents and considerable interactions occur between froth mechanisms (mainly drainage) and processes occurring in the pulp.

(4) The cell residence times for floatable fractions of mineral species have been shown to be an order of magnitude less than those occurring for non floating (gangue etc) mineral species in many flotations. This indicates that the pulp phase cannot be considered as well mixed in terms of the activity leading to the appearance of valuable mineral species at the froth discharge (also froth/pulp interface). Evidence points towards such activity occurring in plug flow plumes through the flotation cell rotor/stator system, rather than the whole tank acting as a continuously stirred reactor vessel.

(5) Three modes of drainage in the froth have been identified as follows:

(a) Film drainage: this occurs as a continuous process in the slurry films between the bubbles, it is most active in the lower (wetter) regions of the froth and applies mainly to water and non-floating mineral species.

(b) Weight induced drainage: this occurs when a given volume of froth matures (due to drainage generally) to such an extent that its weight is too great to be supported and the volume descends to lower levels. This can occur both on a micro-scale (approx 0.5 cm dia) or a macro-scale where large rafts of relatively dry material subduct to lower froth levels. This drainage is in itself non selective, but effectively selective as it occurs close to the froth surface, where the floating minerals are in a majority. It is the principal mechanism returning floatable mineral species to the pulp phase.

(c) Shear induced drainage: this occurs whenever layers of froth move at different velocities and is non-selective as a disturbed froth rapidly drops its load of suspended material. These effects are most active close to the froth discharge where the froth layers accelerate

differently due to the 'free' condition at the discharge lip. The occurrence and effects of this form of drainage have been a major finding in the project work.

The above findings have indicated clearly that the role of rate functions (as considered commonly in the mineral industry) influence, but do not dominate, the response of continuously operated flotation processes. This is likely to cause confusion as the concept of 'fast' and 'slow' floating particles is a good description of the effects noted on operating processes but, in terms of engineering calculations, factors other than flotation rate dominate the production of the apparent rate relationships observed. The flotation model developed at WSL (described previously) utilises flotation rate response, fraction floatable factors and froth efficiency: this blend of factors enabled the model structure to be capable of emulating continuously operated processing plants. However, further progress with models necessitated the inclusion of froth mobility as well as froth drainage factors, froth/pulp interactions and the use of rate functions as descriptions of activity occurring in the rotor/stator region of the pulp phase.

A further model has been developed, based upon the same overall structure as the previous model, where a combined froth and pulp phase approach is used. A combination of finite element and finite domain methods is used to model the mobility and hold up of mineral particles in the froth phase, mechanisms operating in the pulp phase and movements of material across the froth/pulp boundary in both directions.

Further Work

The design of flotation process flowsheets is dominated currently by the use of rules of thumb, empirical relationships and engineering judgements. There appears to be little likelihood of major advances in laboratory-scale testwork methodology and future work on flowsheet design must rely upon accumulated experience. In this light modelling methods with their heavy demands on parameter sets for successful operation must rely also upon the same bank of knowledge for the characterisation of the parameter sets. Hitherto methods of organising knowledge have not been available generally. The advent of knowledge based systems (expert systems) technology [illegible] a vehicle whereby such knowledge banks can be used to generate [illegible] the parameter sets needed

by the process models. The union of [illegible] systems with the modelling methods presents a means of overcoming many of the problems encountered now in flowsheet development in this area.

2

DEPARTMENT OF TRADE AND INDUSTRY

Warren Spring Laboratory, Mineral Processing Division

March 1985: Project MSM 084; Development of a Simulation Package for Mineral Processing Plants

Introduction and Background

A large number of mathematical models and process simulation methods had been developed at WSL over a number of years. These were written for specific purposes and particular applications. This project aims to rationalise and restructure the models and techniques into a package format and develop further modelling methods to complement the existing range. A Prototype package (MIN-SIM) was developed for use as a means of both testing industrial response to the modelling methods and gaining experience from which further developments of the package would evolve. This package contained some inconsistencies and oddities but, in general, the package was well received by the users. A good compromise between user friendliness and engineering content was achieved. The main criticisms centred around the need for graphic display methods and more comprehensive data editing facilities. The use of graphics had been minimised as these systems are computer specific and the writing and debugging of successful (and flamboyant) displays is laborious and time consuming. Data editing is another difficult area; this has been limited in the package format as most computer systems offer good editing facilities and emulating such systems in the package is again laborious and time consuming.

Progress

Subsequent to the trials with the prototype MIN-SIM package and further industrial contacts in the general areas of process simulation and computer hardware development, the general needs and future directions for this project were reviewed. The principle points and guidelines are described and discussed below:

(1) MIN-SIM was developed in the mini computer environment; today such computers have evolved to such an extent that they can be considered as medium sized and, as such, these machines have replaced the smaller 'main frame' machines. Computers in the micro class (personal computer) are

evolving to replace the original concept of the mini class and it is these machines which are now being installed on a general and widespread basis. A medium sized micro (PC) installation is capable of running large sections of the MIN-SIM package. To meet the expected demand for MIN-SIM software in this environment, many of the MIN-SIM programs are being transferred to the PC range in the PC-DOS.2 operating system.

(2) The provision of software packages for the PC range has gravitated to the supply of programs on floppy disks together with support documentation, licencing agreements, and limited back up support. It is clear that direct industrial exploitation of MIN-SIM in the PC environment must follow the general trends for software supply and sales will be in the form of packaged products for particular purposes. A start has been made upon the development of such products. However, the product concept necessitates considerable attention to marketing, documentation and technical support; these activities take time and reduce the effort available for development work.

(3) The prototype MIN-SIM package contains three distinct classes of techniques; a set of materials balancing programs for process investigation, a set of unit process models for detailed investigation of areas of the overall process flowsheet (e.g. flotation processes, grinding installations etc) and general purpose linking models to investigate the whole or larger areas of the flowsheet at a lower level of fidelity. The various unit processes in an overall flowsheet implement operations upon the assemblage of mineral particles in the feed streams in response to various and wide ranging properties of the mineral particles. This wide range of primary mechanisms presents considerable difficulties when considering overall flowsheet simulation methods in a detailed mode of operation. The structures needed to link the unit processes are cumbersome and costly in terms of computational requirements. Detailed overall modelling methods are likely to be big and costly to implement easily ; widespread use of such methods cannot be envisaged in the short term. This view was endorsed by the attendees at the MIN-SIM trials where the major interest was upon better understanding of unit processes and overall flowsheet prediction was considered to be limited mainly to the needs of flowstream and plant sizing, e.g. solids and water balances.

(4) The feedstock preparation section of a mineral processing flowsheet (washing, screening, crushing, ore grinding etc) can, however, be treated as a whole operation with a minimum of difficulty as the operations are all involved with responses to particle size effects. A general purpose modelling method is under development for process 'front ends'. This system uses a general purpose solver (developed and proved as part of the MIN-SIM package for overall flowsheet simulation) as a vehicle for both the linking structure and model developments. The comminution models include both particle breakage effects, selective breakage by mineral species and ore liberation effects.

(5) Simulation in the mineral processing field is limited by a general lack of data in numerically descriptive formats. The major problem is the general lack of adequate and pertinent laboratory-scale testing techniques to provide descriptions of representative ore samples in relation to the behaviour of the mineral ores in production-scale processes. Gravity, magnetic and electrostatic separation methods can be described more readily from laboratory-scale testwork. However, although important locally, these methods represent only a small fraction of the minerals processing scene and before process simulation can achieve a considerable improvement from todays provision it will be necessary to overcome the data problems. Although good numerical descriptions of process activity are the exception rather than the rule, a wealth of process data exists in professional judgement and experience formats. This data could be utilised by the emerging methods of knowledge base engineering and programming (expert systems) to generate the missing numerical data. These systems are in their infancy, but show great promise; some initial work is being undertaken in this area.

Next Period and Further Work

Work will continue on the transfer of MIN-SIM routines onto personal computers in the DOS.2 environment. Further products will be developed both for general use and commercial exploitation. Development will continue on the 'front end' simulation technique, together with the generation of sub-routines to provide the data sets for the models used in the techniques. Further preliminary investigations will be carried out into the likely use and application of

knowledge based systems. Work has commenced on the development of models of crushing plant and thickening/dewatering processes, together with a range of sensitivity analysis methods for MIN-SIM techniques.

1834-1984

Université
Libre de
Bruxelles

Avenue F.D. Roosevelt 50 B - 1050 Bruxelles
Tél. : 02/649.00.30 - Télex 230 69 unilib b

3 Panou

UNIT OPERATION MODELLING IN ORE PROCESSING

Introduction

The results presented here are a summary of our first annual report recently submitted to EEC. The first year of research was entirely devoted to unit operations modelling in ore processing considered on an independant basis. The following operations have been particularly investigated :

- communition
- liberation analysis
- gravimetric separations
- magnetic separations
- flotation

For each of these unit operations a critical synthesis of the existing bibliography has been made. After the adoption of a suitable mathematical formalism and with the help of several minerals and ores of very different nature we have tried to check the proposed formulae and the necessary approximations requested to solve the equations.

Communition

To study the evalution of grinding we have adopted the technique of mass conservation and balance. To solve the integro-differential equations, an hypothesis must be made concerning the selection function : it cannot depend on the environment, i.e. on the nature and size distribution of the other minerals present in the mill.

This hypothesis is rather an over-approximation. To verify it, we have made some laboratory tests with quartz, chromite and magnetite in which the granulometric fractions have been made discernable by either considering the fraction of greatest granulometry or by a substitution of the largest grains than those studied by grains of another mineral easily separated from the former. As an example, during an experiment with quartz, we have replaced the quartz grains of a larger dimension than the granulometric fraction under consideration by particle of magnetite at constant volume or weight.

Figure 2.6.6.3.

For the curves of this slide, we have studied the evolution of 10-14 mesh fraction by considering this fraction either

- alone
- with smaller grains than those of 10-14 mesh
- with smaller and greater grains than those of 10-14 mesh

Of course, the selection function is no longer uniquely determined without specifying the environment

Figure 2.6.7.14.

Here is an exemple of a selection function for the 3 to 4 mesh fraction when the grains belonging to the 6 to 8 mesh interval are replaced by equivalent magnetite grains.

Figure 2.6.7.16.

Another example of a selection function for the same 3 to 4 mesh interval whereby quartz grains of 10 to 14 mesh are replaced by magnetite. As you can see, the selection function is strongly dependant on the environment. This conclusions also apply to the breakage function that is most of the time completely determined as a function of the ratio current size/initial size.

Figure 2.6.8.1.

However, in certain special cases, the breakage function may also depend more directly on the initial dimension, as on the following figure :

Figure 2.6.8.1.

Here we have an example of a breakage function for schist cubes when the sollicitation is acting perpendicular to the schistosity.

Liberation

In the field of liberation analysis, we have first carefully examined the various formalisms adopted in the scientific and technical litterature.
Up to now these formalisms can only be applied to very simple and limited cases.
On the other hand, we have also adopted two different methods to estimate the liberation grade :

- either by considering the granulo-densimetric curves, that is the evolution of different densimetric fractions as a function of granulometry
- or by numbering under the microscope large quantities of grains after a granulometric and densimetric classification.

In the first case and if differential grinding does not play an important role, the evolution of granulo-densimetric curves can give us reliable results on liberation grade estimation.

Figure 3.2.3.12.

For the rare earth ore of the Gakara Mine in Burundi made of bastnaesite and monazite, we can see that the 2.85 - 3.31 densimetric fraction is regularly decreasing for finer and finer grains. This fraction necessarly contains mixed grains as the gangue minerals are almost entirely composed of quartz.
For other types of ores however, the interpretation of granulo-densimetric curves is not so readily simple.
The numbering of particles under the microscope is much more longer but it gives more precise information, because you can estimate the numeral grade of liberation.

Figure 3.2.4.6.

For example, here is the evolution of the mineral liberation grade for stibnite from Groenland which is mainly a scheelite ore, for which of course, similar grade curves exist.
However, when the ore grade is very low, even the measurements under the microscope do not allow the correct recognition of fines inclusions of valuable mineral. This situation result in an over-evaluation of the liberation grade.

Figure 3.2.4.1.

For example, in the case of the pegmatite from the MANONO mine in Zaïre, whose content in cassiterite is about 1,25 kg per ton, the fragmentation gives more than 95% of light material, even for the very fine granulometry. The counting of grains smaller than 65 mesh under the microscope gives a liberation grade greater than 90%. However the washibility curves have further shown that the light grains with a specific gravity less than 2,85 still contained about 25% of the total amount of tin in the form of inclusions hardly or completely invisible under the microscope.

Gravimetric Separations

For gravimetric separations, we have adopted the theory of washability curves. This is not really a modelling of gravimetric process, but modelling is not our main purpose. What we want is to find a way to predict the results of a separation and for this purpose the washability curves are an excellent prediction tool.
The actual experience of the Ore Dressing and Coal Preparation Laboratory of the Free University of Brussels allows us to have now a large quantity of data about the equivalent imperfection and mean probable deviation of the current gravimetric separations devices, or at least those most frequently used in Central Africa.
The only difficult problem is to extrapolate the washability curves outside the practical range permitted by usual laboratory heavy liquids (methylene iodide, whose specific gravity is 3.31). We have developed with success a method allowing the extrapolation by the use of data coming from liberation analysis under the microscope. For this purpose, we have numbered and ranked mixed grains on the basis of their content in valuable minerals and we have introduced the concept of "fictious high-density fractions" whose composition are calculated from numbered grains under the microscope. So, and for the first time, a link between two different unit operations was clearly established under the form of an interaction grinding/liberation and gravimetric separations.

Of course, if the data coming from the numbering under the microscope are rather rough approximation of the reality, the resulting error has an influence on the fictious high density interval, as in the case of the MANONO ore. However, despite this error, the closure balance equation gave results whose maximum error was 10% in the most unfavorable case, and most of the time, the relative error was around 5%.

Figure 5.7.3.23.

For example, there is the washability curves of a wolframite ore from the NYAKABINGO Mine in RWANDA, for the 65 to 150 mesh fraction as a fuction of the grade in WO_3.
Even if this "fictious density fractions" technique has a subjective background, that can introduce errors, this is a very reliable mean for extrapolating washability curves, and thus allowing the correct prediction of gravimetric separations.
It should be noted at this point that in the second part of our research, already under way, we will compare the data-fitting for washability curves with Chebyshev polynomial approximation or by least square aproximation by polynomials or still by other numerical methods.

Magnetic separations

In our research about magnetic separations, we have followed two different ways :

1) by computing field, and as a rather very simple case, we used a cross belt magnetic separator;
2) by application of the theory of washability curves.

In the former case, the calculation of the magnetic field gives rise to too much complicated mathematical formulae with no practical application to real situation. Moreover, several simplifications introduced may lead to unrealistic predictions in certain circunstances.
On the opposite, the theory of washability curves can be easily adapted to predict the results of magnetic separations for para-and diamagnetic minerals. In certain applications we have investigated the grade evolution of different minerals at the same time.

Figure 6.3.2.8.

For the MAVROLAKKOS iron ore from the island of THASSOS in Greece, in addition to the analysis of the iron concentration by magnetic separators, we have also investigated the eventual production of a baryte concentrate. In other cases, as for a chromite ore coming from CUBA,

Figure 6.3.2.10.

beside a final magnetic clean-up to slighty increase the grade in Cr_2O_3, the main purpose was the increase the Cr/Fe ratio. We have thus drawn on the same figure the washability curves for the Cr_2O_3 grade and the Cr/Fe ratio.

Flotation

About flotation, we have made a clear distinction between continuous and discontinuous flotation.

A. In discontinuous flotation kinetics, all our experiments have shown that, in opposition to certain affirmations, it is vain to describe the kinetics with a specified order.
As fundamental variable we have adopted a parameter called the quality (x) which represents the aptitude to flotation of mineral grains measured by the method of the bubble pick-up test. Even if this technique is contestable, all our experiments have shown that, for any experimental time, the sum of the quality distributions in the sink and float is constant. This certainly introduces an effective reliability in our results.
For examples, there we present a three-dimension diagram

Figure 7.5.4.3.

showing the distribution in time of the quality (x) for a bituminous schist from MOROCCO. With the help of geostatistics, we have proved that this moroccan schist can be concentrated after a moderate grinding at dimensions several times higher than those of liberation. No kinetic order can exist either at constant quality, nor from a global point of view.
In the case of quartz grains flotation with amines,

Figure 7.5.2.6.

we have also noticed that quartz grains with a high aptitude to flotation

have a slower kinetics compared to the first order law. As a result, the global kinetic behaviour of quartz grains (full line) depends on the initial distribution of quality among the quartz grains.
However, if we have a look at the grains that undergo flotation for very short intervals of time, the quality distribution of these grains follow a rather normal law.

Figure 7.5.2.8.

Although the sum of normal distributions is still a normal distribution, the global flotation kinetics does not follow such a law because, once the grains with a ligh aptitude have undergone flotation, the remaining particles do not follow a normal law for these high value of the quality as they have almost disappeared from the flotation call.
Anyway, with the help of these measurements of the aptitude to flotation, one can draw washability curves as well as partition curves for various flotation cells.

Figure 7.5.2.10.

As example, we have represented the washability curves as a function of the aptitude to flotation for the case of pure quartz grains.
Even if these curves have no absolute value, as for gravimetric and magnetic separations, they represent an interesting mean to investigate flotation possibilities and cells efficiency.

B. Regarding continuous flotation we have adopted a very different point of view because, with the exception of small variations, the continuous flotation process is stationary. The composition inside a flotation cell remains thus constant in time, in opposition to what happens in discontinuous flotation.
From this stationarity concept, we have established our own formalism which is quite similar to the one used for our investigations on grinding, more particularly by the introduction of a selection function which is a measure of the flotation probability of a grain during its lifetime in the cell.
It is true however that the practical application of this formalism requests the determination of the flotation or not of a large number of fine grains of equal aptitude to flotation. Therfore it is difficult to develop a current application.

Conclusion

In spite of all the limitations, we believe that it is not necessary at first to overcome all the difficulties in the modelling of each of the unit operations before starting a new step in our investigations.
During the second phase of research that is going on now, we are developing micro-computer software taking account of our know-how and experience in this field but with a special emphasis on the various links between the different unit operation..

Figure 2.6.6.3 : Quartz - Evolution de la tranche granulométrique 14-20 mesh

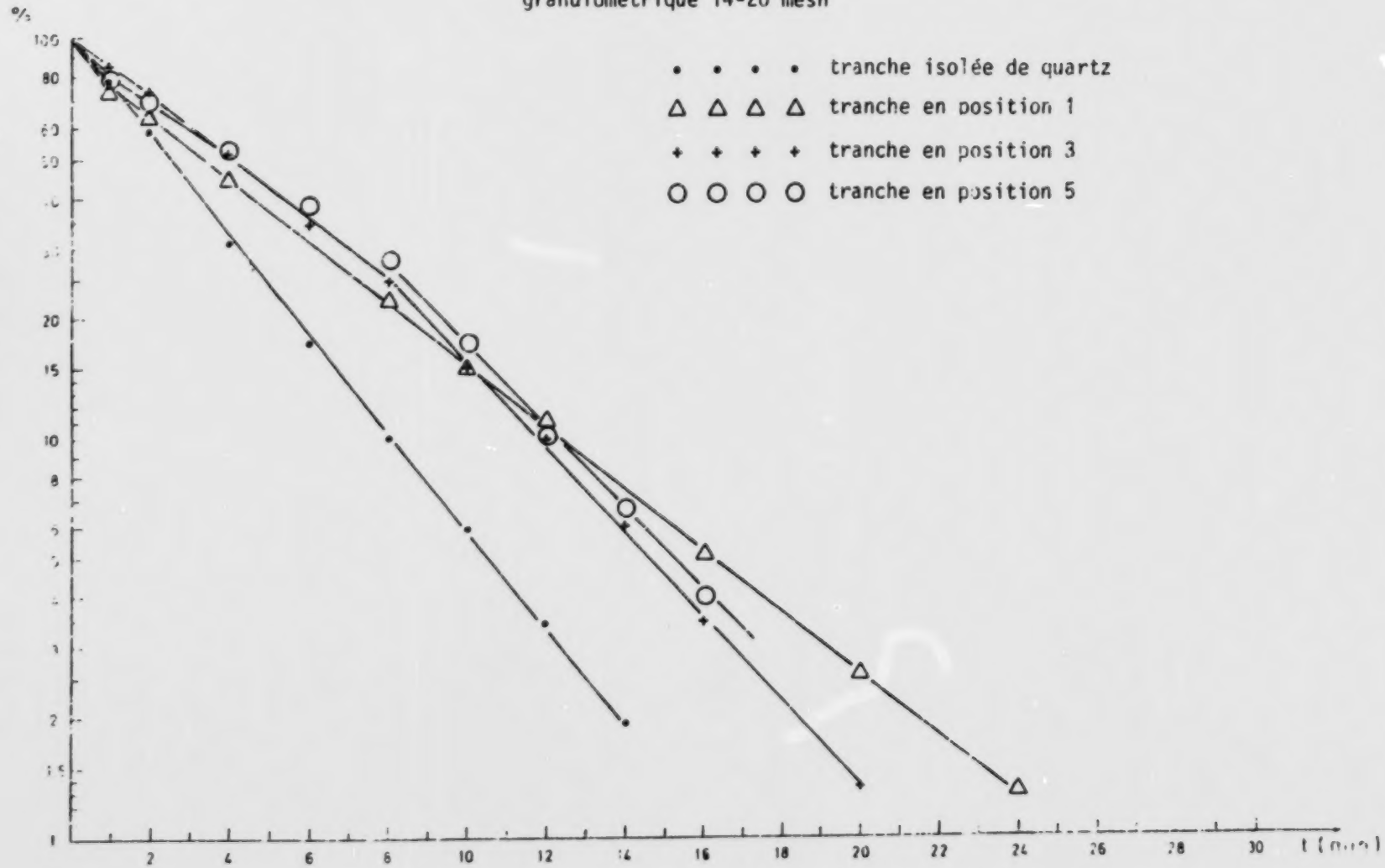


Figure 2.6.7.14

Probabilité de broyage Q (----) et fonction de sélection S (——), en fonction du temps (minutes), de la tranche granulométrique 3-4 mesh

Quartz lorsque la tranche 6-8 mesh a été substituée par de la magnétite.

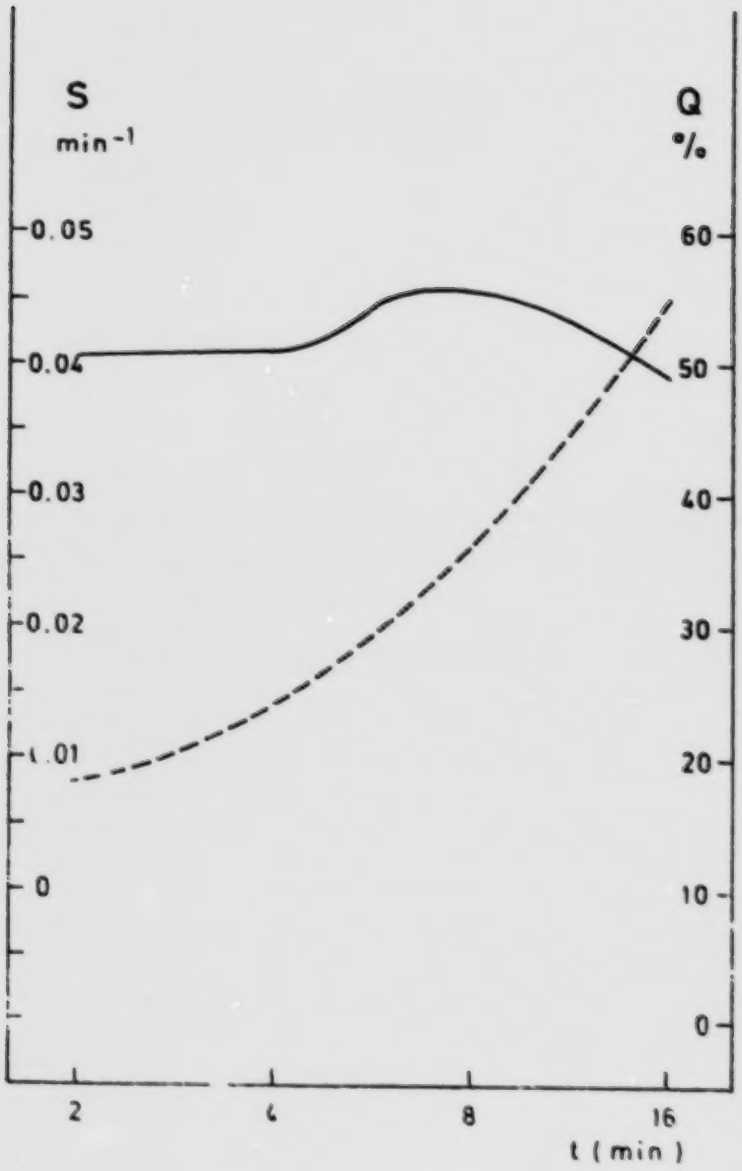
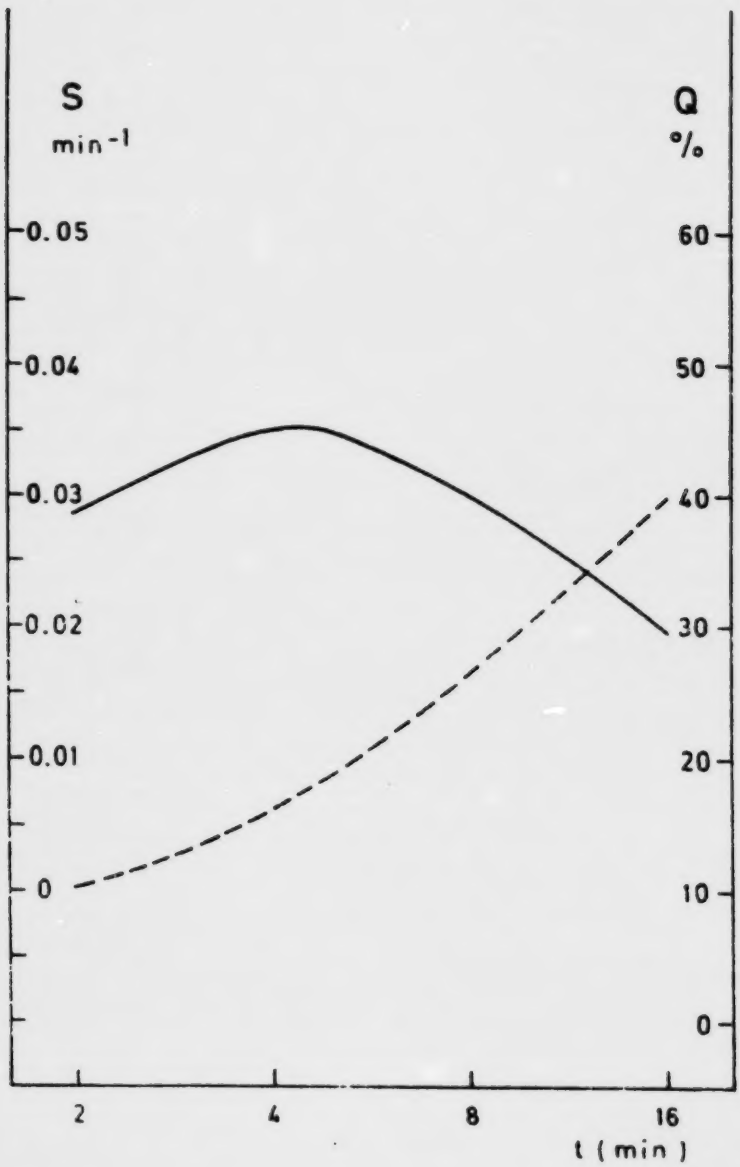


Figure 2.6.7.16

Probabilité de broyage Q (----) et fonction de sélection S (——), en fonction du temps (minutes), de la tranche granulométrique 3 - 4 mesh.

Quartz lorsque la tranche 10-14 mesh a été substituée par de la magnétite.



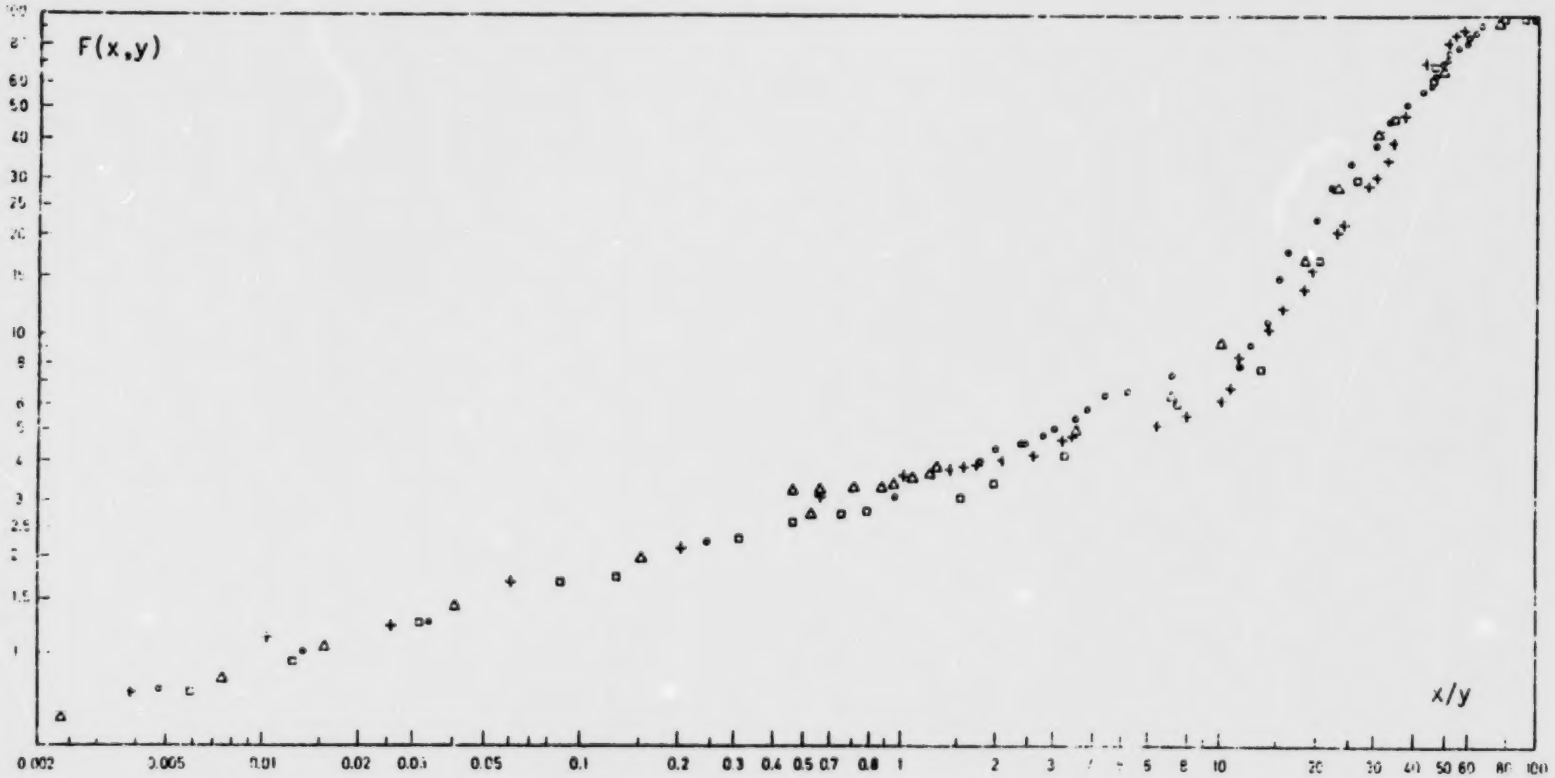


Figure 2.6.8.1. Détermination expérimentale de la fonction F(x,y) pour des galets de quartz concassés à la presse

- • grains dont le poids moyen est 26,9 gr
- + grains dont le poids moyen est 38,4 gr
- △ grains dont le poids moyen est 54,9 gr
- □ grains dont le poids moyen est 74,7 gr

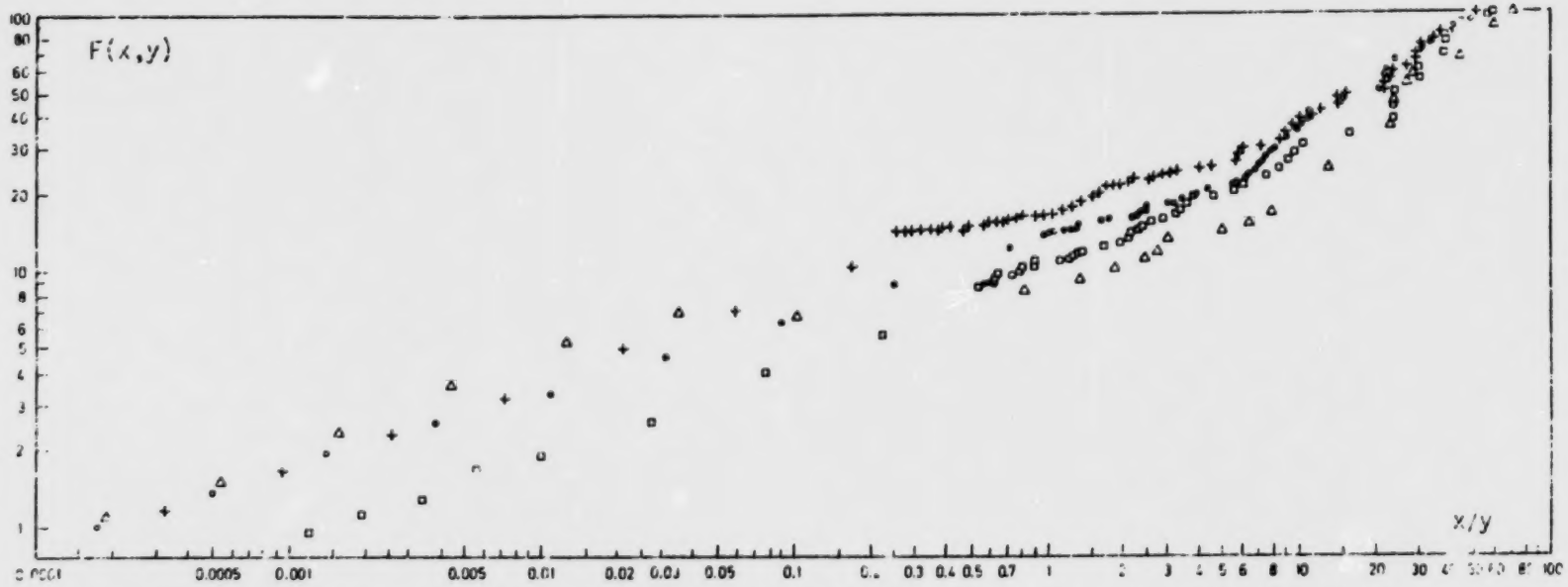


Figure 2.6.8.3. Détermination expérimentale de la fonction F(x,y) pour des cubes des schiste ardoisier sollicités perpendiculairement à la schistosité.

- grains dont le poids moyen est 63,2 gr
- \+ grains dont le poids moyen est 95,5 gr
- △ grains dont le poids moyen est 128,1 gr
- □ grains dont le poids moyen est 202,4 gr

Figure 3.2.3.12. Courbes granulodensimétriques du minerai de GAKARA 1.

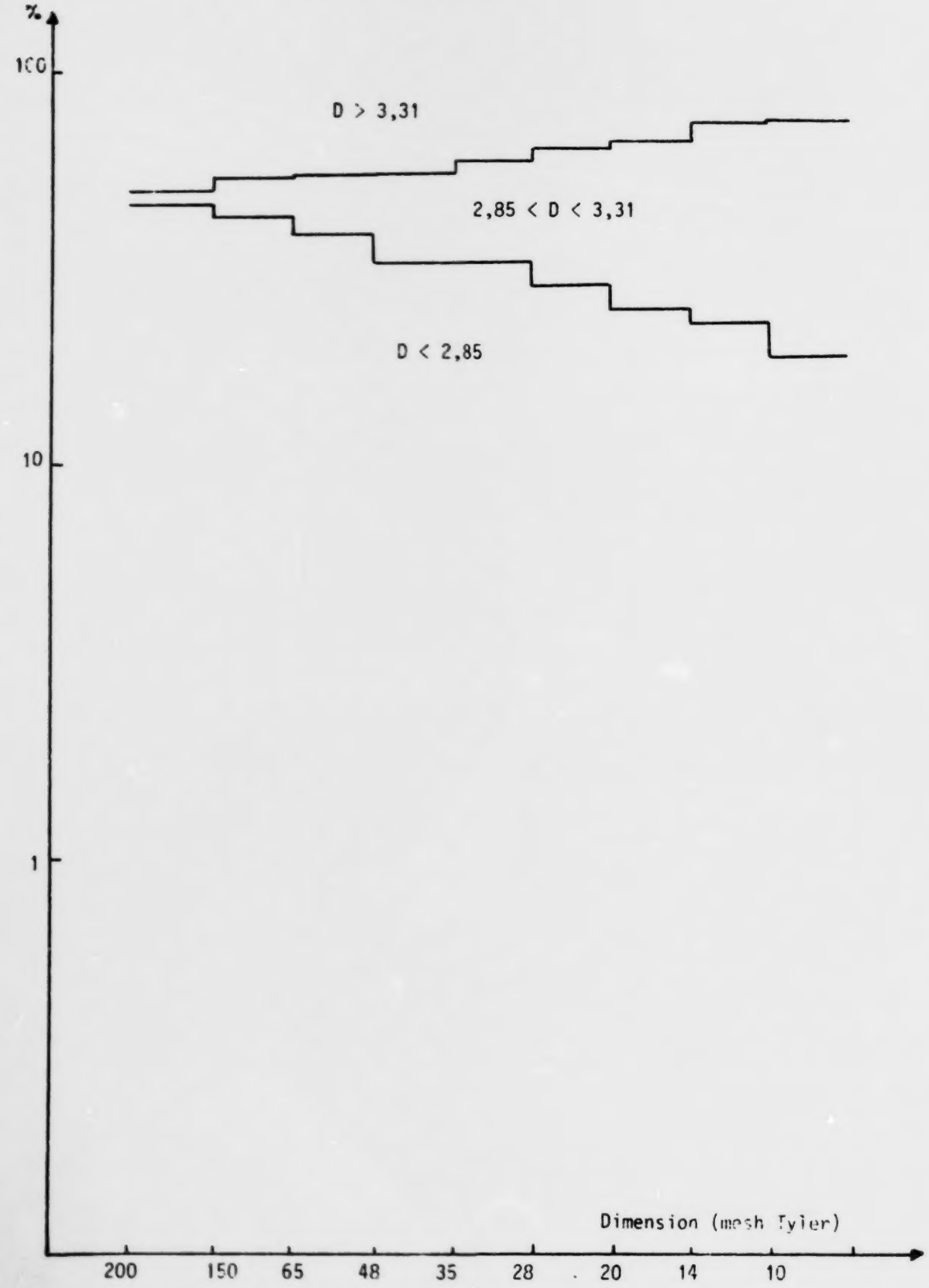


Figure 3.2.4.6

Evolution du degré numéral de libération de la stibine.
Minerai de Margeries Dal Nord.

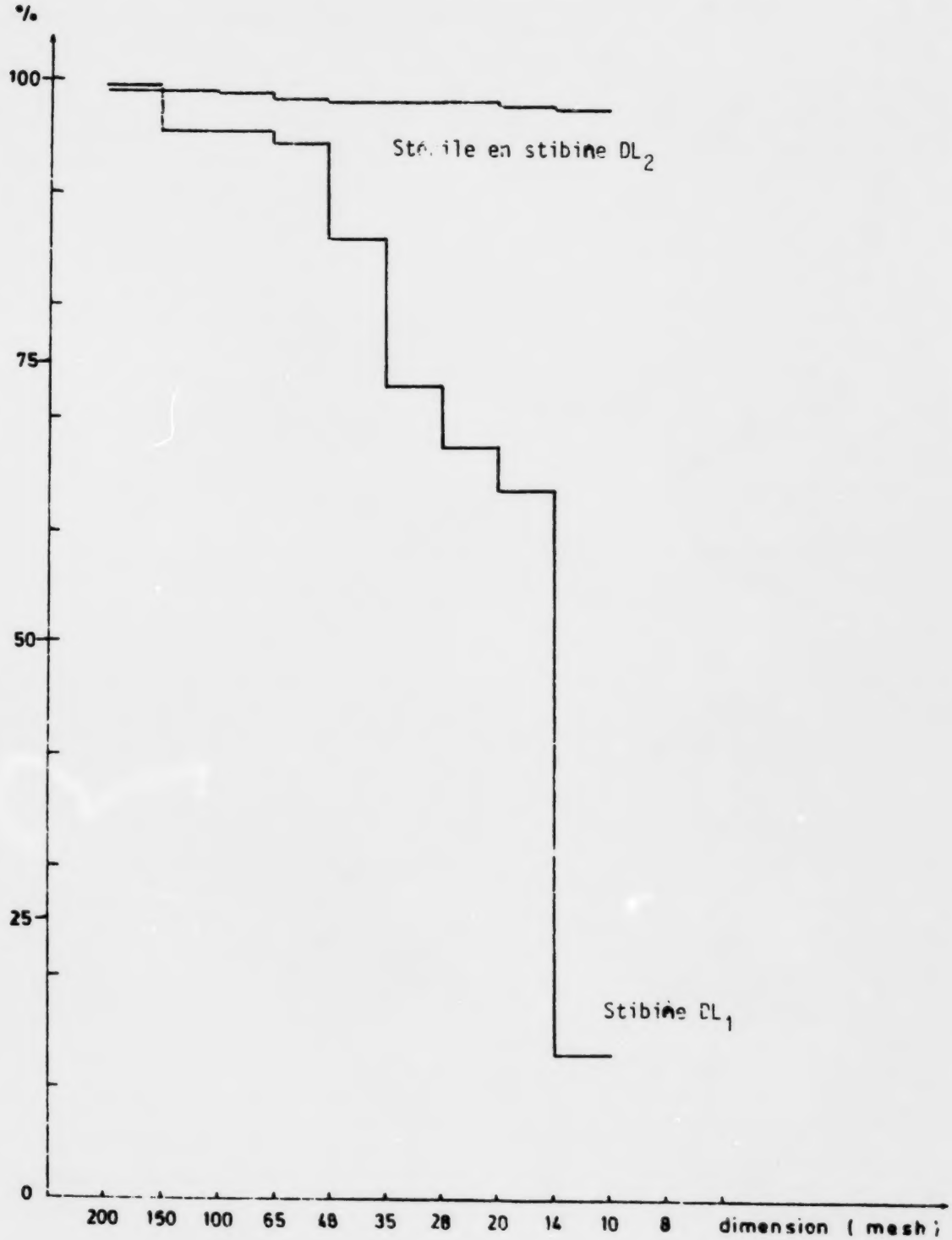


FIGURE 5.7.3.23.

Minerai de NYAKABINGO I - 65-150 mesh

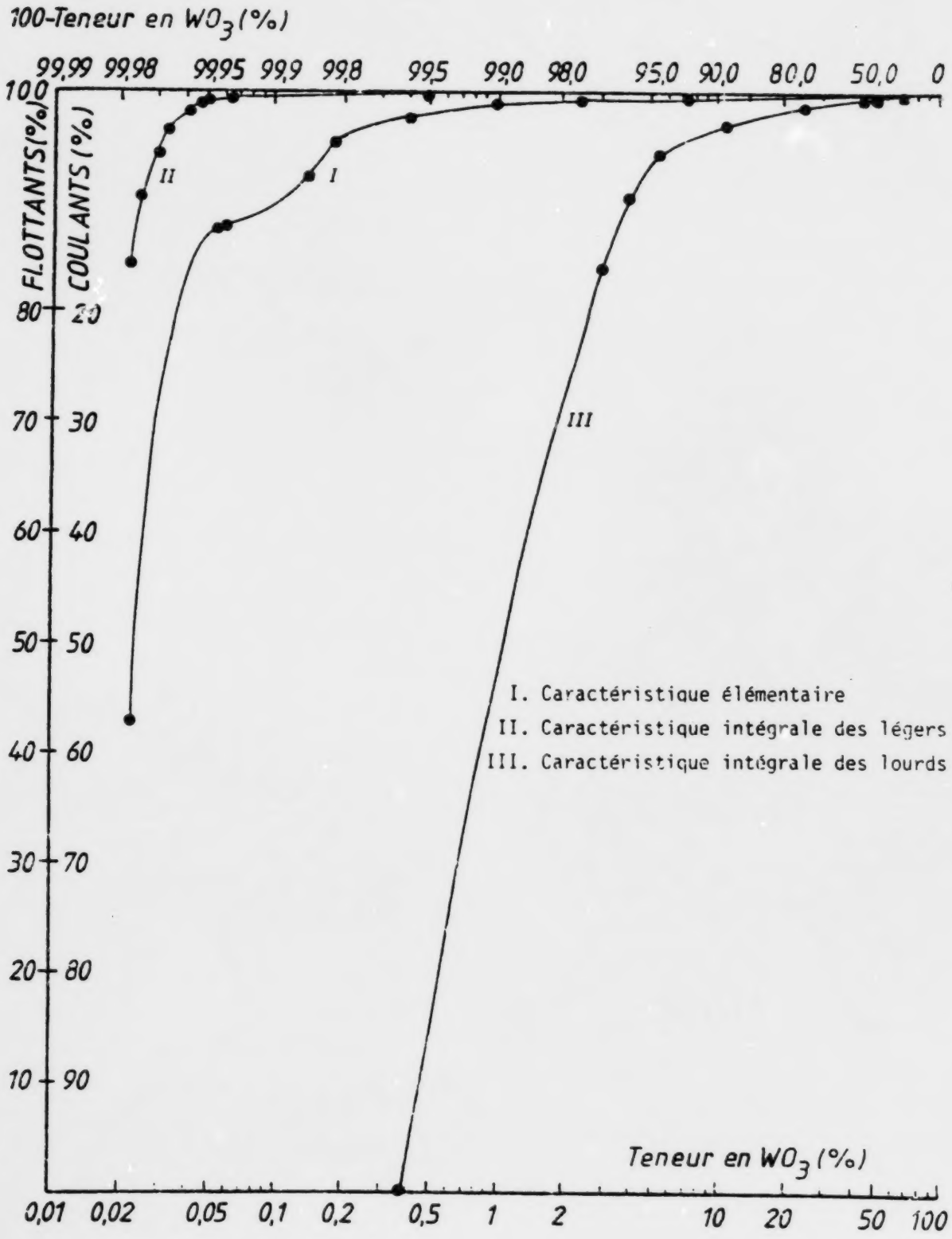
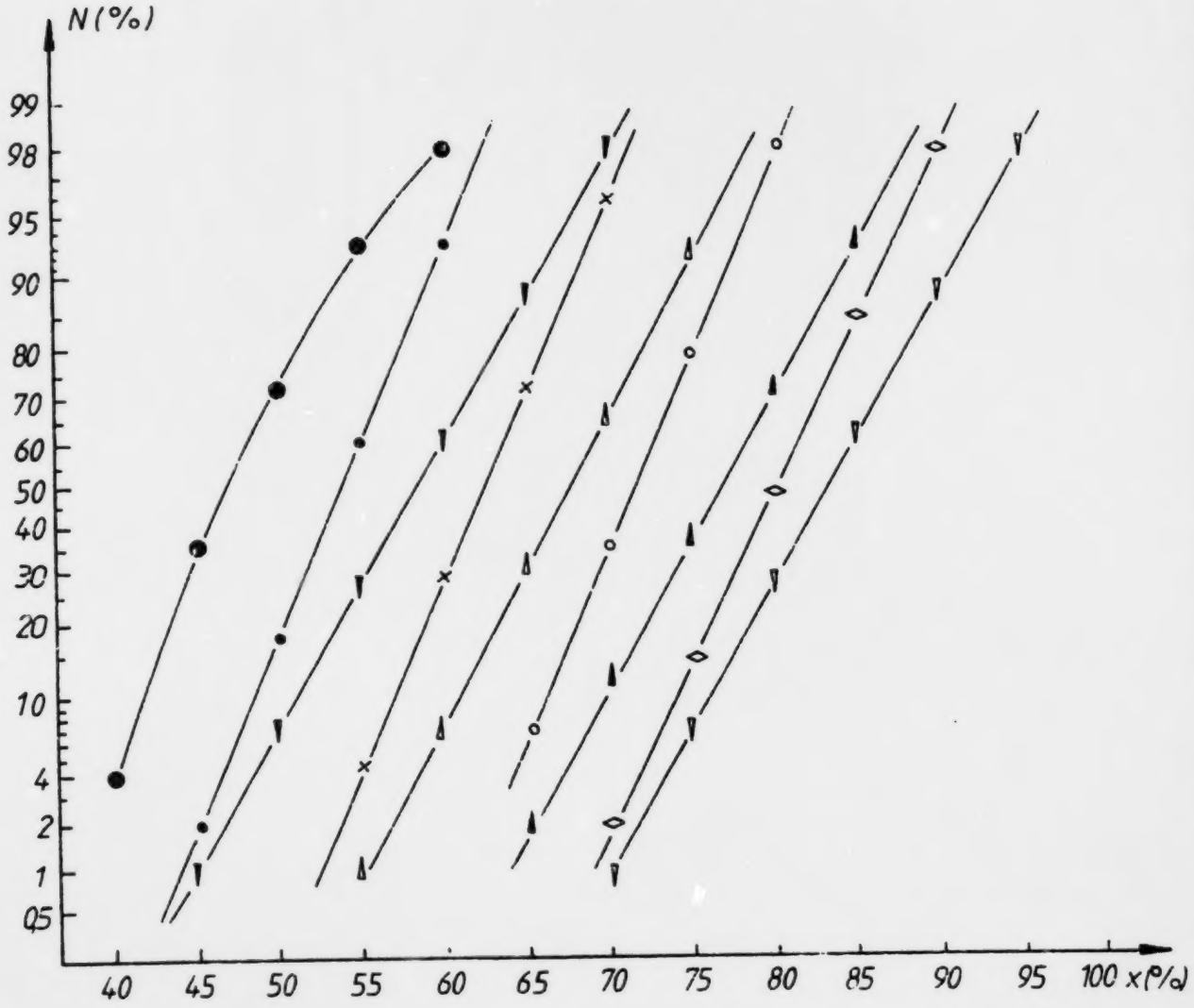


FIGURE 7.3.2.8.

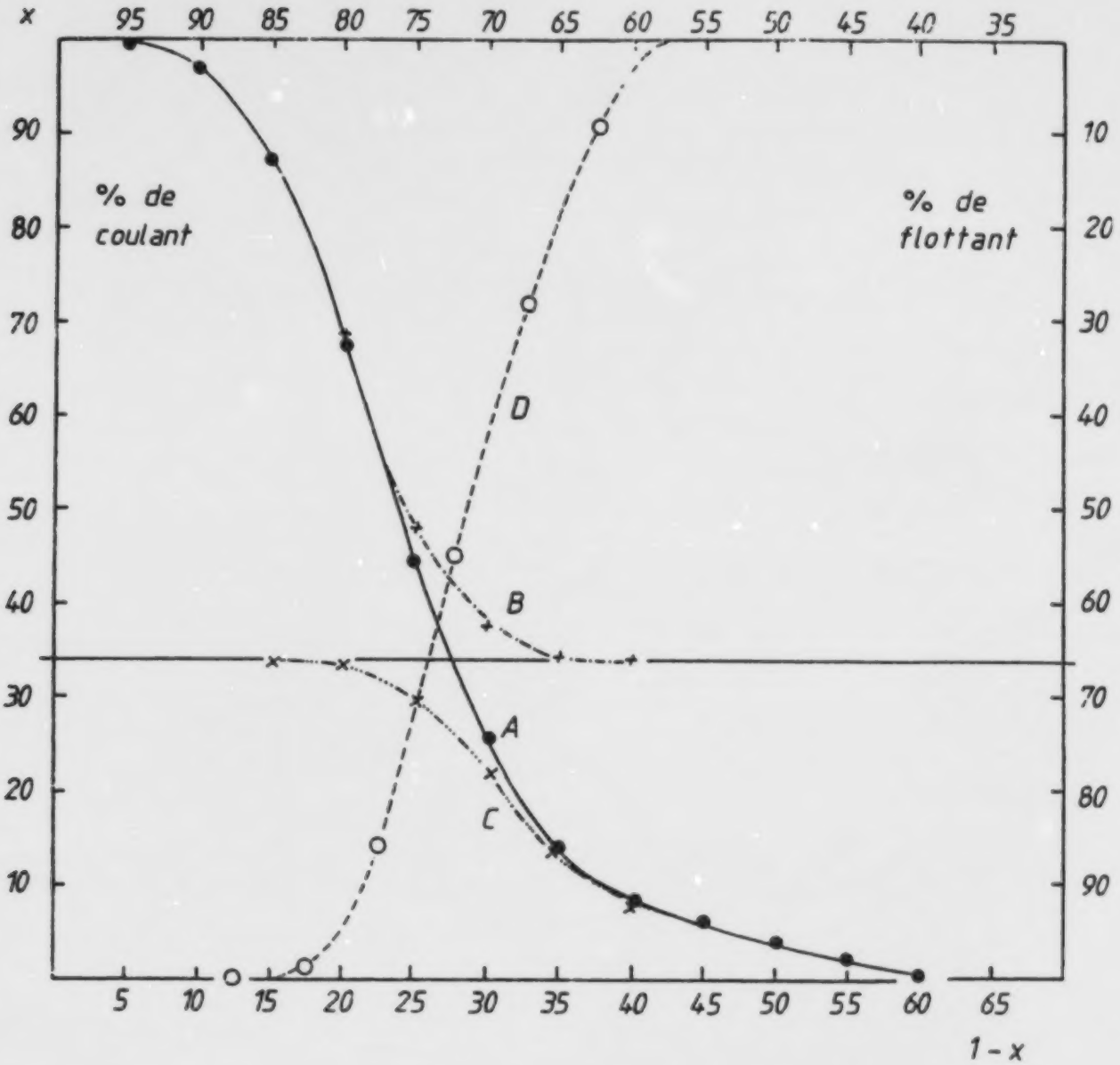
Distribution de la qualité dans les flottants partiels.



- intervalle de temps de flottation 0-6 s
- intervalle de temps de flottation 6-10 s
- intervalle de temps de flottation 10-20 s
- intervalle de temps de flottation 20-25 s
- intervalle de temps de flottation 25-60 s
- intervalle de temps de flottation 60-75 s
- intervalle de temps de flottation 75-110 s
- intervalle de temps de flottation 110-135 s
- coulant après 135 s

Figure 7.3.2.10.

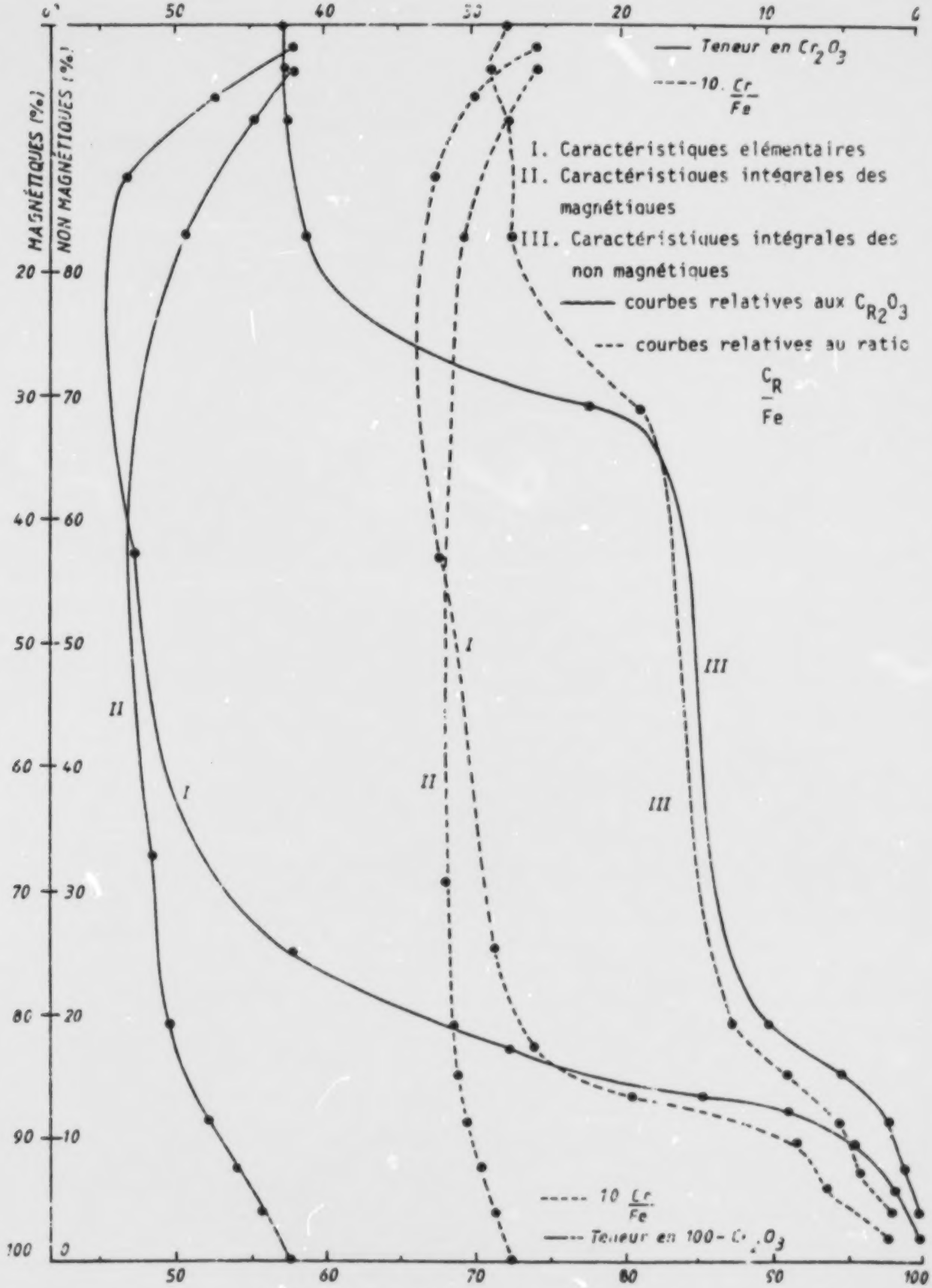
Représentation d'une flottation de quartz par les courbes de lavabilité en fonction de la qualité. Tranche granulométrique 150-200 mesh. Temps de flottation 20 s.



A : courbe de lavabilité de l'alimentation
B : courbe de lavabilité du flottant
C : courbe de lavabilité du coulant
D : courbe de partage

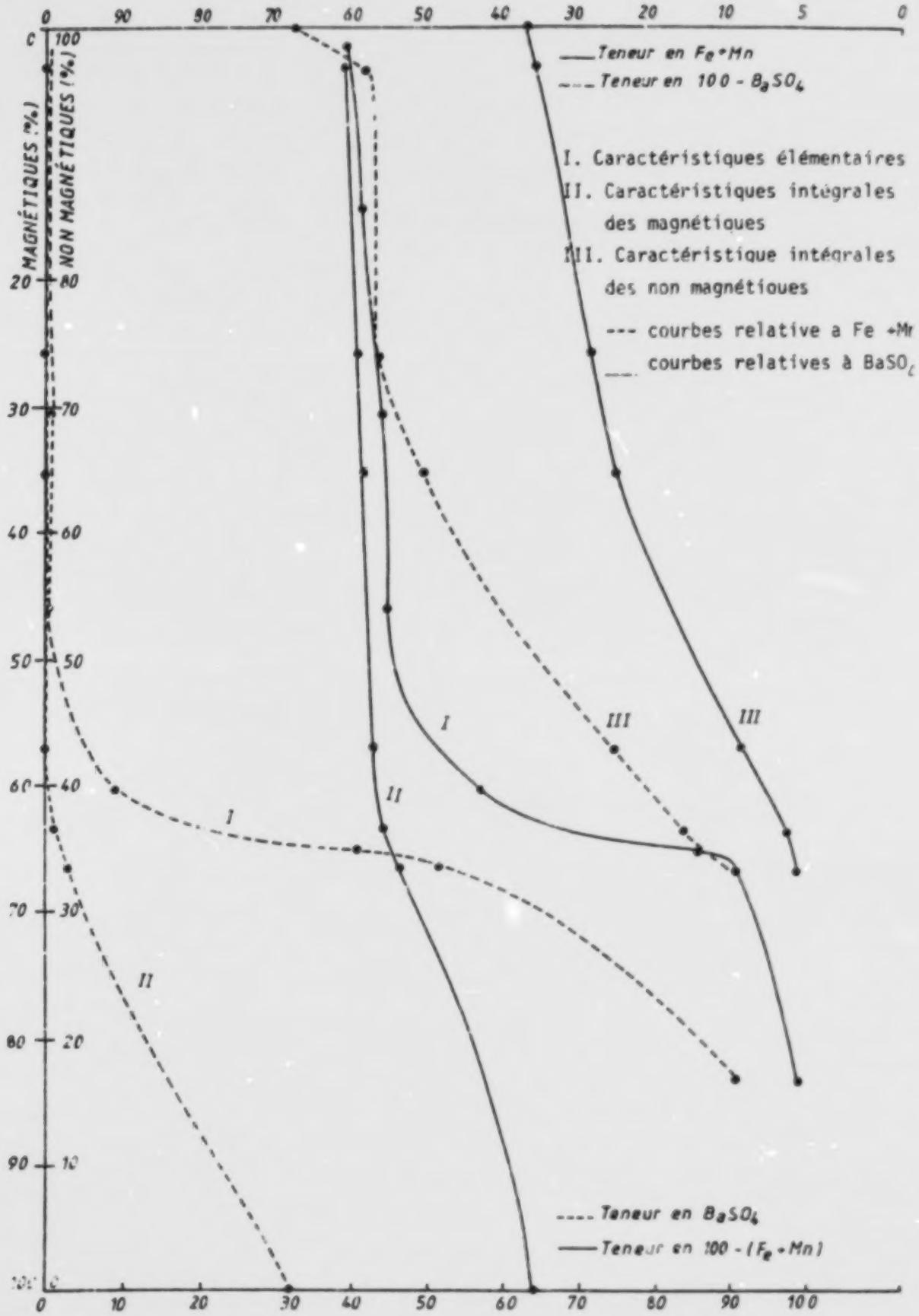
Figure 6.3.2.10. : Minerai de Chromite de CUBA

35-48 mesh



MINERAI DE MAVROLAKKOS

20-25 mesh


MAGNÉTIQUES (%)
NON MAGNÉTIQUES (%)
Teneur en Fe+Mn
Teneur en 100 - BaSO4
I. Caractéristiques élémentaires
II. Caractéristiques intégrales des magnétiques
III. Caractéristique intégrales des non magnétiques
--- courbes relative a Fe +Mn
___ courbes relatives à BaSO4
Teneur en BaSO4
Teneur en 100 - (Fe+Mn)

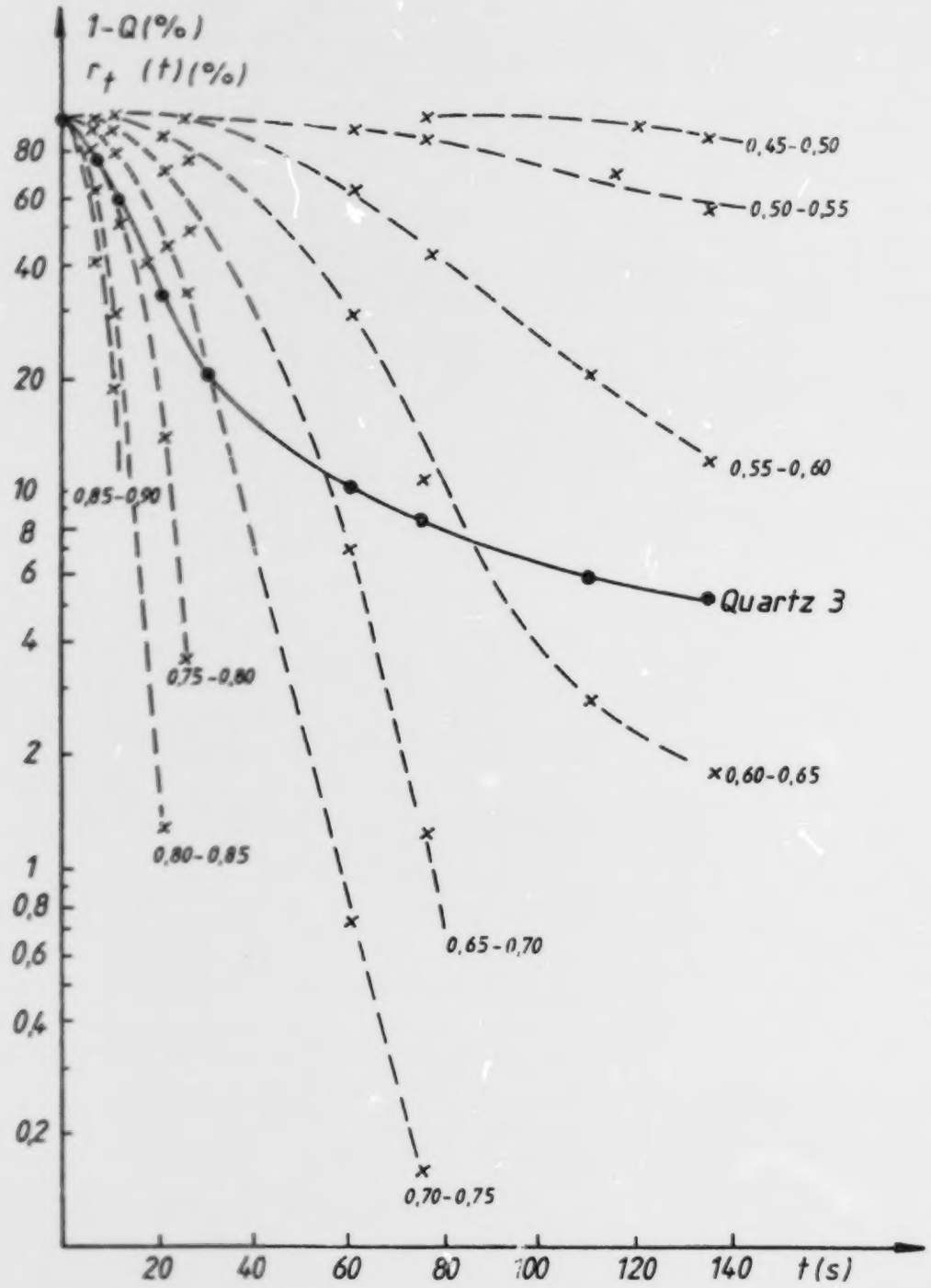


FIGURE 7.5.2.6.

Variation de la probabilité de non-flottation et du rendement pondéral global du coulant en fonction du temps.

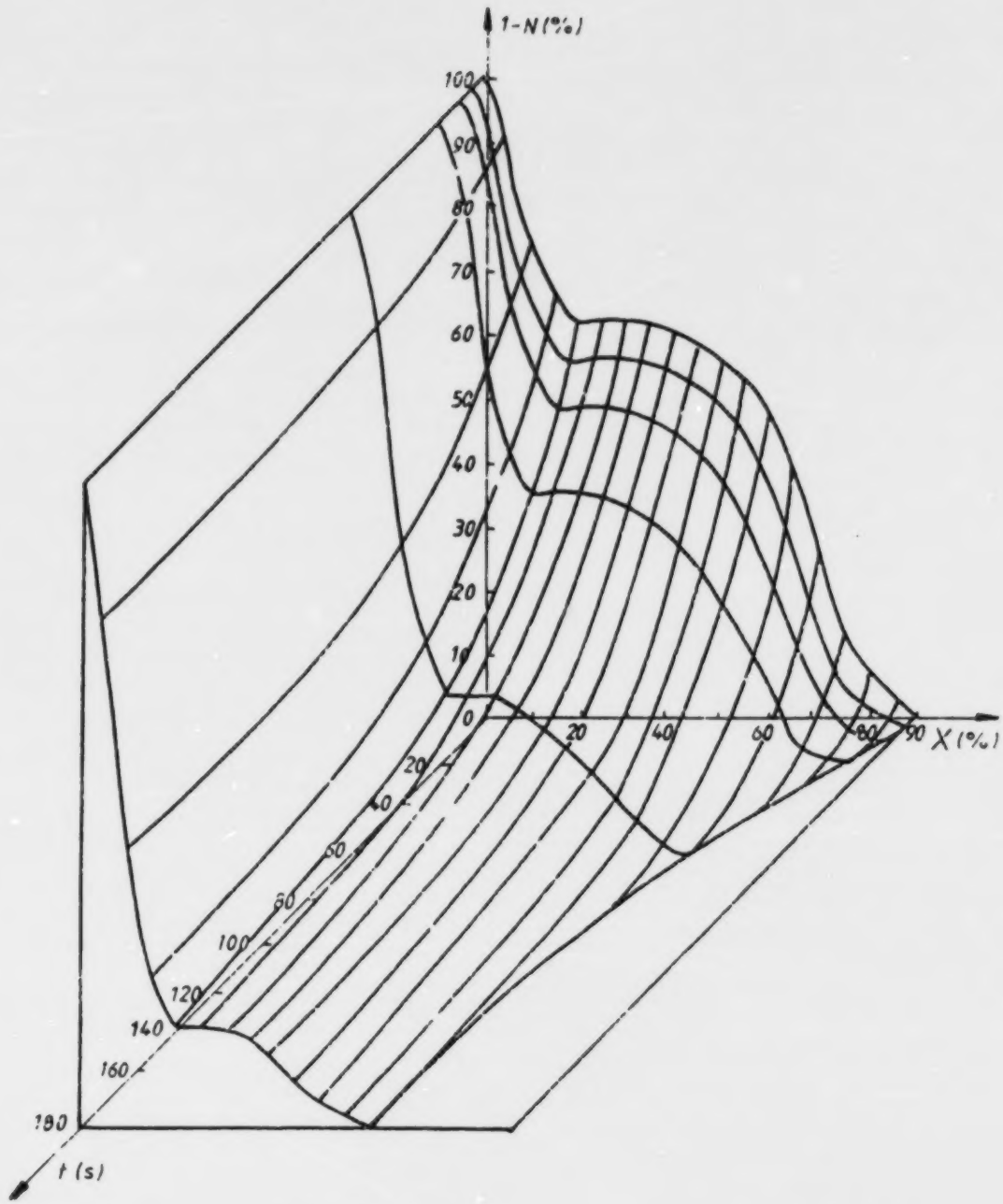


FIGURE 7.5.4.3.

Courbes de distribution de la qualité du shiste bitumineux broyé à 100 % sous 100 mesh (147 µm).

4

REUNION CEE/CANMET des 13-14 mai, 1985

Remarques préalables du BRGM

3 domaines de recherche scientifique sont concernés par la réunion:

1 - la libération minérale et le contrôle en minéralurgie

2 - la technologie des fines particules, essentiellement flottation de minerais sulfurés fins complexes

3 - le contrôle des ions Fe en hydrométallurgie

- Sur le troisième domaine, le BRGM n'a pas d'activité de recherche, ni d'intention de recherche, actuellement.
- Sur le deuxième domaine, le BRGM ne bénéficie pas actuellement d'aide de la CEE. Il s'agit cependant d'un des domaines de compétance du BRGM, sur lequel il est très actif depuis plusieurs années.

Les points forts du BRGM sont notamment:

- la possession d'un pilote récent instrumenté de broyage-flottation d'une capacité de 250 à 450 kg/h selon les minerais
- une expérience considérable en minéralogie qualitative et quantitative des minerais sulfurés complexes. Le BRGM a été amené à réaliser des travaux pour d'importantes sociétés étrangères spécialisées dans les sulfurés complexes. - une activité permanente de mise au point de procédés en laboratoire,

- le BRGM a actuellement plusieurs cibles importantes en cours d'étude.

Des propositions seront vraisemblablement soumises à la CEE si le cadre d'un prochain appel d'offres le permet et alors des échanges d'informations scientifiques avec la partie canadienne pourront être envisagés.

- Sur le premier domaine couvert par la réunion, la libération minérale et le contrôle en minéralurgie, le BRGM bénéficie d'un contrat en cours avec la CEE.

1. Libération minérale

1.1. Eléments sur nos recherches récentes

L'objectif de notre dernieère phase de recherche était d'améliorer notre méthodologie d'interprétation des résultats des mesures à 1 Dimension et 2 Dimensions au moyen de l'analyseur d'images. La démarche a comporté successivement des simulations par ordinateur, une réflextion théorique, et des mesures réelles sur un minerai sulfuré.

Les simulations conduites sur des populations de particules sphériques de morphologie et de mixité variées ont permi de dégager des considérations importantes sur:

- la richesse des informations issues de différentes méthodes de mesure en absence de toute méthodologie d'interprétation 3D. Notamment l'intérêt des mesures 2D limitées à la zone équitoriale de la particule a été mis en évidence à cause de la ressemblance de ces mesures avec la réalité 3D.
- le nombre souhaitable de grains à observer pour avoir une information précise.

Mais surtout, ces simulations ont conduit à suggérer puis à démontrer une nouvelle méthode d'évaluation de la proportion de particules réellement libérées, qui reposent sur l'utilisation simultanée de mesures 1D et 2D.

Soit PB la proportion de particules de phase B réellement libérée.

En général PB est inconnu, les mesures par analyse d'image 1D et 2D conduisent à deux estimateurs biaisés par excès de PB : PBL et PBS

$$PBL > PBS > PB$$

PBL et PBS sont respectivement les proportions de particules apparemment libérées de phase B observées à 1D et à 2D.

Nous avons demontré qu'il existe un autre estimateur, également biaisé par excès mais meilleur que les précédents:

$$PBL > PBS > 2PBS-PBL > PB$$

et souvent $2PBS-PBL = Pb$

Les mesures réelles sur minerai sulfuré ont notamment confirmé la possibilité d'utilisation pratique de cette méthode. Elles ont aussi permis de comparer cette méthode avec des approches totalement empiriques, et de constater une concordance rassurante.

1.2. Possibilité de coopération avec les chercheurs canadiens

- une relation de coopération scientifique existe déjà entre le BRGM et l'Université Laval, Québec, concrétisée en avril 1985 par une publication commune G. Barbery (Laval) A. Broussaud (BRGM).

2. Contrôle des usines en minéralurgie

2.1. Résumé des recherches récentes du BRGM

2.1.1. - Les recherches conduites par le BRGM en collaboration avec la société européenne "Barytine de Chaillac" feront l'objet d'une présentation au congrès international de minéralurgie de Cannes (France) en juin 1985.

Un échantillonnage ponctuel de l'usine de broyage flottation a été suivi du traitement des données numériques issues de l'échantillonnage par un logiciel de bilans matière pour établir un bilan détaillé cohérent pour 8 tranches granulométriques et 5 constituants chimiques de l'usine. Ce bilan détaillé cohérent a été utilisé pour construire le modèle de simulation de l'usine. L'influence des conditions d'alimentation de l'usine sur les résultats prévus par simulation a été comparée aux tendances mises en évidence par une étude statistique des relevés quotidiens de production. La bonne concordance constatée a permis de valider le modèle et de l'utiliser pour simuler différents flowsheets et proposer des modifications de flowsheet de l'usine. Une démonstration a ainsi été faite que la simulation, même avec des modèles simples permet d'apporter une aide effective à l'optimisation ou à la conception d'usines minéralurgiques.

2.1.2 Plus récemment nos recherches se sont orientées vers l'inventaire des modèles simples développés dans le monde et convenablement décrits dans la littérature technique, et le test de ces modèles par confrontation à des données industrielles. Par modèles simples nous entendons modèles qui peuvent être utilisés dans un logiciel de simulation d'usine après mesure d'un nombre restreint de paramètres.

2.1.3 Intentions de recherche pour l'avenir

2.1.3.1 - Tout d'abord nous souhaitons poursuivre l'inventaire et le test industriel des modèles simples, en couvrant successivement les différentes opérations unitaires de minéralurgie: broyage, classification granulométrique, flottation, filtration... Cette action se situe dans la perspective de développement de la conception d'usines minéralurgiques assistée par ordinateur (C.A.O.).

2.1.3.2 - Ensuite, nous souhaitons contribuer au développement de la conduite par ordinateur des concentrateurs, qui se justifie par les progrès récents dans:

- la fiabilité des capteurs
- la modélisation des procédés
- le coût du matériel informatique.

Nous envisageons deux sujets de recherche dans cette optique:

- le développement de capteurs nouveaux permettant de mesurer certains paramètres de la phase liquide dans les procédés de flottation, en particulier de minerais sulfurés. Une aide de la CEE pourrait être solicitée pour les prochaines années sur ce sujet.

- le développement d'outils informatiques (logiciels) destinés à être implantés auprès des opérateurs des concentrateurs, pour leur fournir en temps réel:
  - des bilans matière des concentrateurs
  - des conseils de réglage de l'usine.

  Il s'agit là d'une étape qui nous paraît raisonnable si ce n'est indispensable sur le chemin de l'automatisme intégral.

2.2 Possibilités de coopération avec les chercheurs canadiens

Au-delà des relations déjà établies avec CANMET et l'Université Laval, nous sommes intéressés à priori par des actions qui nous permettraient de tester des outils nouveaux (logiciels, capteurs) sur des cibles industrielles. Il pourrait s'agir par exemple de tester des outils développés au Canada sur les données dont nous disposons auprès de nos partenaires industriels actuels, ou inversement d'essayer de résoudre des problèmes de l'industrie canadienne avec les méthodes maitrisées par le BRGM.

I

Jones

AUTOMATIC METHODS FOR CHARACTERISING THE MINERALOGY OF LOW-GRADE AND COMPLEX ORES

Among the mineralogical features that must be measured to characterise ores and mineral products are:

1. the compositions and the textures of rocks,
2. the identifications, the locations, and the sizes of mineral grains, and
3. the sizes, shapes, compositions and textures of mineral particles.

Experience has shown that it is often difficult to obtain adequate optical contrast to achieve automatic discrimination of the various minerals in a specimen. Consequently, the research has been directed at non-optical methods of analysis - in particular, the use of signals which are produced when a specimen is bombarded by a finely-focussed electron beam. These signals include those derived from x-rays and from back-scattered electrons. The x-ray signals can be analysed either by wavelength dispersion methods or by energy dispersive analysis: the back-scattered electron signal can be collected either by an annular detector or by using segmented detectors. In these ways, discrimination of minerals can be based on the use of numerous independent (but simultaneous) signals.

Our analytical system uses an electron probe x-ray microanalyser which is fitted with 4 wavelength dispersive spectrometers as well as a segmented back-scattered electron collector.
The x-ray signals can provide excellent mineral discrimination but they provide much slower information than optical and back-scattered electron signals. Consequently, the x-ray signals are only used for LINEAR analyses whereas the other signals can also be used for AREAL analyses.

In the LINEAR method the mineral specimen is "stepped" beneath a focussed electron beam. It takes about 10 ms to establish which mineral is under the beam. The specimen is then moved forward so that a contiguous "point" on the specimen surface can be analysed.

The X and the Y co-ordinates of the specimen are recorded whenever the electron beam crosses onto a new mineral phase. These co-ordinate values are used to calculate the lengths of the intercepts which are made across the various minerals. The intercept lengths can then be used to determine:

1. mineral proportions and specific surface areas,

2. grain size distributions,

3. ore textures, and

4. particle shapes, compositions, and textures.

There are many sources of ambiguities during both linear and areal analyses; for example, the artefacts which are introduced by specimen preparation procedures, and the uncertainties which frequently occur at grain boundaries. Many of these ambiguities can be removed, either by improving the specimen preparation methods or by using suitable "filtering" algorithms that recognise the artefacts and edges and adjust the analytical results accordingly.

Results from a linear (or from an areal) analysis will, occasionally, apply directly to the 3-D bulk of the measured specimen. More commonly, however, the linear data provide only biassed estimates of three-dimensional characteristics. Ideally, therefore, both linear and areal data should be transformed into their equivalent 3-D values. However, this is rarely possible, as yet, and the MEASURED values are often taken to be equivalent to the true three-dimensional values.

When very low grade ores are being analysed there are special difficulties in locating adequate numbers of the mineral grains of interest. However, the automated microanalyser can be set to search for these grains over large specimen areas.

SUMMARY

The results of x-ray based linear analyses have already been used in the design of mineral separation processes and for assessing the performances of these operations. In addition, the analytical results have been used to assess the amount and kind of liberation achieved during grinding.

The work done, so far, has clearly shown the feasibility of carrying out AUTOMATIC linear analyses using x-rays to differentiate the various minerals. The back-scattered electron signals are now being incorporated into the mineral discrimination algorithm and will, eventually, significantly reduce the time taken for some kinds of analyses.

SESSION II

Fine Particle Technology

Canada - EEC Meeting - May 13, 1985

Fine Particle Recovery

Last year's workshop in Brussels covered a wide range of topics in the fine particle recovery topic. For the benefit of those who were not there, let me review quickly what was discussed.

1) Mr. George Marizot of the Bureau de Recherches Geologiques et Minieres of Orleans, France, described the work underway on gravity separation and flotation of complex sulphide ores. The performance of gravity equipment is being assessed for material 100 m and physico-chemical parameters will be evaluated. In flotation, adsorption and desorption phenomena are being studied and an on-line sensor is being developed to measure collector concentrations.

2) Mr. A. Kremser then discussed the flotation improvements implemented at the Meggan Mines high pyrite Pb-Zn circuit. The Meggen ore is known for its extremely fine dissemination of galena, sphalerite and pyrite. The paper outlined the better grades of pyrite obtained for acid production and the rejection of thallium and mercury for environmental requirements.

3) Dr. F. Ricalens of Minemet Recherche (Imetal) talked about the planned pilot studies in a new plant which is being built. These studies will be done on complex fine grained ores trying to minimize the production of ultrafine particles, to determine the best flotation parameters for different collectors and to optimize these parameters to obtain salable, selective products. As usual, gelena is the problem mineral.

4) Dr. J. Bessière of the Centre de Recherche sur la Volorization des Minerais of Nancy gave a resume of their work on ionic flotation in acid medium. Previous work touched on the beneficiation of uranium present in phosphoric acid and the elimination of cadmium again from phosphoric acids. Present studies are concentrating on molybdenum beneficiation and separation from sulphuric acid leaching of uranium, more work on cadmium elimination from phosphoric acid production and the elmination of chrome from industrial effluents is also being carried out.

5) Mr. L. Vollbracht, from ENKA Research in Arnhem, discussed the possibility of using and the production of selective flocculants to recover fine particles escaping recovery by flotation. This talk covered reagents derived from polysaccharides and their characteristics. Short chain polysaccharides have dispersing properties whereas long chain polysaccharides have flocculating properties. Dispersing properties can be improved by incorporating anionic groups. Selective adsorption of the polymer is the key to its selective application. No hard data on selectivity and recovery were given.

6) Dr. Kocabay of Imperial College, London, described the application of new recovery techniques to the separation of fine mineral particles. The recovery techniques being investigated are all based on surface properties and include microbubble flotation, selective flocculation, spherical agglomeration and two-liquid flotation. The ores under study are phosphates with calcareous gangue, complex sulphide ores, tin ores and tungsten ores. Preliminary results indicate that two-liquid flotation and spherical agglomeration have possibilities.

7) The last contractor to speak was Dr. P. Parsonage of Warren Spring Laboratory. He discussed in general terms some of the progress in the prediction of flocculation behavour from electrokinetic measurements and in the choice of reagents which may be of potential use in selective flocculation. Molecular structure on complexing and dispersing ability of the reagents appears to be the governing factors. Two novel techniques for fine particle separation were mentioned. One is the selective magnetic staining in which one mineral is coated with magnetite, then separated magnetically and the other one is continuous electrophoresis which is a method used in biochemistry to separate cell components.

CANMET has been involved in ore treatment for a good many years and has gained a lot of experience in handling problems of all kinds including fine particle recovery as a part of the total package treatment. It is recognized that more effort is required to deal directly and specifically with fine particle recovery and a new project was created to deal exclusively with this topic.

Both oxides and sulphides will be incorporated in the project. The types of materials we intend to deal with at the present include tin minerals lost in base metal flotation. (Brunswick Mining and Smelting, Cominco, Kidd Creek), tantalum fines from Tanco (Ta O). Lead-Zinc flotation tailings from Pine Point will be used for the base metal studies.

The chemical-mineralogical characteristics of the various minerals are being investigated by size fractions down to 1 or 2 m. Besides the surface studies, equipment and procedures will also be looked at. Under consideration are the Nagahm cell, an electroflotation and microfoam flotation cells of our own design, selective flocculation, new flotation reagents and sulphide depression systems.

To conclude, a few words on possible future research. Somasundaran in his Gandin Lecture of last year summarized, very neatly, basic avenues which we should also consider as being of the utmost importance. Mass and increases in surface energy and surface area were always though to be the major causes for the difficulties in separating fines. Alterations in morphology, mineralogy and surface chemical

composition with decrease in size should also be considered as responsable for the different behaviour of mineral fines.

I will now turn the meeting over to _____ so that we can hear from the _____ contractors on their past year's work.

MINEMET RECHERCHE

société anonyme capital 8 000 000 f rcs Versailles 301608634 b siret 301608634 00012

1, avenue Albert Einstein b.p. 106-ZJ. 78191 Trappes Cedex-Téléphone et Télécopieur (3) 050.61.88

télex Mitec 696745 F

Dpt Minéralogie-Minéralurgie
68.85.042 JJP/jv

J.J. PREDALI

Trappes, le 13 février 1985

Monsieur DONATO

C/C M. RICALENS

OBJET : Projet de sujet de collaboration C.E.E. - Canada

Problèmes posés par la flottation des fines particules de sulfures

Situation du sujet

Dans les minerais sulfurés à gangue essentiellement pyriteuse et à maille de libération très fine, la récupération des métaux nobles est insuffisante ; elle décroît généralement dans l'ordre suivant : Zn, Cu, Pb et Ag. Ce phénomène est d'autant plus marqué que les particules sont plus fines et plus fragiles (cas de la galène).

Un deuxième phénomène négatif apparaît aussi : la moindre sélectivité observée dans les concentrés monométalliques, particulièrement les entrainements plomb dar le concentré de cuivre et cuivre dans le concentré de plomb (1).

Les recherches antérieures entreprises par Minemet Recherche ont eu pour but de déterminer si ces résultats étaient dûs à la taille des particules, ou à d'autres phénomènes. Les résultats obtenus (2)(3) ont montré clairement que les particules de sulfures purs flottent parfaitement même dans les fractions les plus fines (0-4 microns). Par contre, lors de mélanges synthétiques avec de la pyrite, la galène fine flotte très mal, alors qu'en mélange avec le quartz le phénomène n'apparaît pas.

Les problèmes de mauvaise flottabilité paraissent donc bien provenir des interactions entre particules de sulfures et pyrite, et tout particulièrement des phénomènes se produisant lors du broyage. L'action poursuivie par Minemet Recherche donc été orientée vers l'étude des conditions de broyage et de la recherche de réactifs modificateurs.

Certains résultats positifs ont été obtenus au niveau des mélanges de sulfures, par contre, l'application à des minerais se heurte à de nombreuses difficultés qu'il est nécessaire de dépasser par un prolongement des travaux.

Références :

(1) JJ PREDALI - JPh BEUVELET : Problèmes posés par le passage du laboratoire au pilote lors de la flottation des minerais complexes.
XVème Congrès International de Minéralurgie - Cannes 1985

(2) JJ PREDALI - JPh BEUVELET : Contrat avec le C.E.E. n° MPP-132-F - 1982

(3) JJ PREDALI et al. : Spectroscopic study of potassium amylxanthate adsorption on finely ground galena : relations with flotation.
Congrès I.M.M. - Rome 1984

AINEMET RECHERCHE
68.85.042 JJP/jv

(2)

Projet de programme

1/ Meilleure connaissance des états de surface, notamment de la galène finement broyée, au moyen des méthodes spectroscopiques.

2/ Maitrise des conditions de broyage au stade laboratoire, afin :

a) de diminuer la production de particules ultrafines,
b) de diminuer les interactions sulfures-pyrite,

par contrôle du potentiel d'oxydo-réduction et des ions en solution.

Recherche de modificateurs chimiques de contrôle du potentiel.

3/ Comparaison de différents schémas de flottation :

séquentielle Cu-Pb-Zn, semi-différentielle Pb+Cu suivie de séparation Cu/Pb.

- Recherche de collecteurs spécifiques du cuivre par rapport à Pb et Zn ;
- Recherche de déprimants spécifiques moins polluants que NaCN et SO_2 et ses dérivés.

Applications : à différents minerais Cu-Pb-Zn-Ag et Cu-Zn-Ag pour améliorer la récupération des métaux.

Un effort particulier sera mené pour augmenter la récupération de l'argent contenu dans les sulfo-sels (tétraédrite, ...) à maille de libération très fine qui sont habituellement perdus dans la pyrite ou flottent avec la blende et ne sont pas valorisés dans le concentré de zinc.

****

FINE PARTICLE PROCESSING

P.Parsonage
Mineral Processing Division
Warren Spring Laboratory
Gunnels Wood Rd.
Stevenage, Herts., U.K., SG1 2BX
Telex:82250

The relevant projects being carried out at Warren Spring concerned with fine particle processing are shown below:

1.Selective flocculation.

2.Surface properties

3.Reagents in solid/liquid systems

4.Reagent club (database)

5.Selective magnetic coating

A brief summary of the scope of the projects is given below.

1.Selective Flocculation. This is a separation technique which has potential application for the recovery of fine mineral particles which are at present untreatable by conventional mineral processing techniques. The objective of the process is to produce conditions in a suspension such that on addition of a flocculant, aggregation of one phase occurs. Separation of the flocculated phase may then take place by settling or elutriation.

In the project recently completed we investigated the chemical and process parameters that influence the efficiency of the selective flocculation process. Specifically we examined:-

i) The effect of zeta potential on flocculation, how zeta potentials can be measured and predicted for mineral-solution systems, how they can be altered and controlled by dissolved species and dispersants, and how polymer adsorption is affected.

ii) Effects of polymer anionicity on flocculation of different minerals.

iii) Effects of co-grinding.

iv) Effects of steel grinding on suspension stability.

v) Selective reagents for control of surface and solution properties. We investigated reagent structures suitable for complexing cations in solution whilst having little tendency to adsorb at the mineral surface, and conversely dispersants with low complexing ability.

Techniques used for the investigation include:
Zeta potential measurement using laser-Doppler velocimetry.
Aggregate size analysis and size variation during flocculation using laser (Fraunhofer) diffraction
Electrode potential measurements
Stability tests
Computerized methods for predicting ionic species in solution.

The applicability of selective flocculation has been assessed, mainly in relation to systems containing hematite, cassiterite and wolframite. Our conclusions are that the process is most appropriate where the phase to be flocculated is a substantial proportion of the feed as is the case in the beneficiation of iron-ore where the hematite forms about 50% of the feed, and a pilot-plant operation is currently being undertaken at Wadi Sawawin in Saudi Arabia using selective flocculation prior to flotation based on the work done at WSL. When lower grade feeds are treated such as tin or tungsten ore cyclone overflows, we have found that in order to obtain satisfactory recovery, grades must be sacrificed and the potential for using the method to treat these types of material does not appear to be great.

Of particular concern in fine particle systems is the effect of slight changes in ionic composition of the pulp on the overall system behaviour; this has been shown to be of great importance in the iron-ore example referred to earlier. Changes in the calcium ion concentration due to changes in feed or water quality drastically affect the flocculation behaviour of the hematite and it has been found that the dosage of dispersant must be controlled within close limits for the process to work efficiently.

These aspects of interaction between reagents, minerals and ions in suspension have been studied more explicitly in our "Surface Properties" project.

2.Surface Properties. The scope of this project is to investigate the effects of reagents on the surface properties of particles in suspension as a means of aiding methods of separation. Specifically we are looking at the following:

i) The influence of collectors on the dynamic surface tensions of solutions.

ii) The developement of contact angles on pure mineral plates as a function of time, reagent concentration, and pH. Initial tests have been with fluorite, calcite, and magnetite in the presence of oleic acid, calcium ions and depressants.

iii) Measurements of solubility products and equilibrium constants of collectors with various metal cations.

iv) The effects of reagent structure on the adsorption of reagents and the subsequent effects on the system properties. The diagram shows a summary of some of this work where we investigated the effects of reagent structure on their effectiveness as dispersants for cassiterite. In addition we have studied the effects of structure on complexing ability for various cations in suspension.

v) New Reagents. Investigation of new reagents of the imino methylene phosphonic acid type and their applications to selective flotation.

Overall the surface properties project is aimed at providing a ratioal basis for reagent selection.

3.Reagents in solid/liquid systems. Control of bulk suspension properties is the theme of this project which has recently started. Investigations will be carried out to study the influence of surface properties on the handling characteristics of slurries, with particular reference to:

i) Dispersion and aggregation

ii) Rheology and flow properties

iii)Filtration and dewatering

iv) Foaming and defoaming

4.Reagent Club (Database). This project is a computerized database we are constructing containing information on reagent usage and properties. Eventually it will contain information relating to flotation practice worldwide, cross-referenced with fundamental chemical data such as solubility products and stability constants of reagents with various metal cations. In this way we will have rapid access to current flotation practice in a way not possible at present.

5.Selective Magnetic Coating. This process has been developed at WSL for the separation of difficult ores. It relies on the adsorption of colloidal magnetite onto selected particles which can then be recovered by conventional magnetic separation. Efficient separations have been obtained with mixtures of carbonate/ apatite, beryl/ silicate, -20 micron cassiterite/ quartz, and metallic lead/ copper. Selectivity is obtained by control of the surface properties of the particles in the suspension. Plans for pilot-plant testwork are in hand.

Overall Theme of Projects. The overall theme for the current

projects is the function of reagents in controlling suspension properties and in helping in the selection of reagents for specific purposes. Initially we are not studying specific systems or types of separation but are looking at fundamental aspects of surface modification, thus providing the broad base that is required when dealing with complex systems such as are found in mineral processing systems.

Our findings could most effectively be applied in collaboration with other workers in the development of processes for the treatment of specific ores.

RECHERCHES DE BASE CONCERNANT LA VALORISATION DES MINERAIS SULFURES COMPLEXES FINEMENT DISSEMINES PAR FLOTTATION DIFFERENTIELLE : LES CONDITIONS D'UNE RECUPERATION OPTIMALE DE PLOMB

J.M. CASES, M. KONGOLO, A. BURNEAU, L. MICHOT, J.J. PREDALI

Au cours d'un précèdent contrat on a montré que les causes de la mauvaise récupération de plomb devaient être recherchées par ordre d'importance dans les phénomènes suivants :

a) Slime-coating.

b) Mixité souvent intime de minéralisation.

c) Difficulté de flotter les fines de galène à des concentrations tolérable

La présente recherche a donc pour objet, d'une part, d'étudier l'origine du slime coating en vue de proposer d'éventuelles solutions, d'autre part, de mettre au point des méthodes expérimentales permettant l'étude des mécanismes de collection dans de bonnes conditions.

1. L'étude du recouvrement des grains grossiers de pyrite par la galène s'est déroulée de la manière suivante :

a) Mise au point d'un broyeur en inox pouvant utiliser des barres inox ou fer et permettant le contrôle, au cours du broyage, du pH, du potentiel d'oxydo-réduction, de la pression partielle d'oxygène. Les produits broyés étant caractérisés par les courbes granulométriques effectuées au Sedigraph 5000.

b) Par des essais de broyage effectués à pH contrôlé, pH 4, pH 7, pH naturel, pH 11 (NaOH et CaO) et pH 12. Dans une première phase ont été broyées les phases pures. Des essais ont en outre été effectués avec le broyeur utilisant les barres de fer à pH naturel. Les produits broyés ont immédiatement été flottés (tube Hallimond en l'absence de moussant). On a immédiatement, après broyage, étudié le comportement électrophorétique des fines ainsi obtenues en traçant deux types de courbe :

- Evolution du potentiel électrocinétique en fonction du pH et de la force ionique (KCl).

- Evolution du potentiel électrocinétique en fonction du pH et de la concentration en collecteur.

Les résultats obtenus sur les phases pures montrent qu'a priori il est possible de limiter fortement le slime-coating en broyant, à pH basique, CaO n'étant pas forcément le meilleur régulateur de pH à utiliser. Ces conditions ne sont pas les meilleures pour obtenir, par la suite en flottation, une bonne sélectivité de la galène vis-à-vis de la pyrite. Les conditions optimales étant grossièrement :

pH au broyage compris entre 7 et 9.

pH de flottation compris entre 9 et 11.

La concentration en collecteur joue un rôle considérable dans un domaine étroit de concentration.

2. L'étude des mécanismes de la collection des sulfures a été entreprise par deux voies différentes :

   - Analyse des produits immédiatement après conditionnement ou flottation par spectrométrie infra-rouge avec ou sans transformée de Fourier. Cette recherche a permis de mettre en évidence sur les fines de galène l'évolution des phases superficielles avec le pH :

     . Sulfate et carbonate de plomb à pH acide ou neutre.

     . Carbonate basique de plomb à pH basique.

     . La présence aux concentrations en collecteur, supérieures à $5 . 10^{-3}$ Ml de xanthate de plomb en couches multiples mélangé dans certains cas au dixanthogène, aux valeurs des concentrations en collecteur voisines de $1 . 10^{-3}$ Ml^{-1}, d'un complexe superficiel - Pb Ax responsable de la collection aux faibles concentrations.

   - Détermination des isothermes expérimentales des collecteurs par chromatographie en phase liquide haute pression.
     Les essais effectués dans un premier temps pour les pressions partielles en oxygène égales à zéro et après nettoyage de la surface de la galène à l'EDTA et à l'alcool de manière à lessiver les phases oxydées ont montré que l'affinité du collecteur pour la surface de la galène était nulle, même pour les concehtrations en tensio-actif de l'ordre de $5 . 10^{-3}$ Ml^{-1}.

     Les essais sont en cours en vue de tester l'influence des états d'oxydation des surfaces sur la collection.

SESSION III

Iron Control and Elimination in Hydrometallurgy

CANADA/EEC MEETING ON THE PROCESSING OF COMPLEX MINERALS - MAY 13, 1985

IRON CONTROL IN HYDROMETALLURGY

by

J.E. Dutrizac

By way of introduction, it should be noted that iron is the fourth most abundant element in the earth's crust, and as such, it is a common constituent in virtually all ores and concentrates. It is especially prevalent in complex sulphide ores where it creates problems in all parts of the processing cycle. The iron contained in complex sulphide ores can be conveniently divided into two general categories based on its mineralogy, and these two categories are illustrated in Figure 1.

The first category consists of those minerals which have iron as an integral part of the lattice of the sought-after minerals themselves. For example, sphalerites from Canadian complex sulphide ores typically contain 5-10% Fe substituting for zinc, and of course, chalcopyrite contains major amounts of iron. Tetrahedrite is an important silver carrier in many complex sulphide ores and this mineral often contains several percent iron. It is important to emphasize that these significant concentrations of iron are contained in the sought-after minerals themselves, and this iron cannot be removed however efficient the grinding and flotation practices may be.

In complex sulphide ores, the iron problem is further aggrevated by the initimate association of the sought-after minerals with other iron-rich minerals, notably pyrite. Pyrite is the matrix, as it were, for many Zn-Pb-Cu ores. In theory, the pyrite can be removed by selective flotation, but in practice there are real economic limits to the amounts of iron sulphide which can be eliminated. The upshot of these two categories of iron is that selective concentrates or bulk concentrates produced from complex sulphide ores are invariably iron-rich. Iron is a reactive metal which is quite soluble in acid media. Hence, if hydrometallurgical methods are employed for the processing of complex sulphide ores, a large amount of iron invariably reports to the solution. Because the amounts of dissolved iron are so large, bleed streams cannot normally be used for iron control, and at some point the iron must be precipitated. It is here that the real difficulties begin because the iron must be precipitated economically and in a form which allows easy filtration and efficient washing.

As concerns operational or rapidly developing technologies relative to the iron control problem, it should be noted that a number of Canadian companies are currently involved with processing the concentrates made from complex sulphide ores, and these organizations have developed procedures to handle the associated high iron contents. Both Canadian Electrolytic Zinc and Kidd Creek Mines process conventional zinc concentrates derived in large part from complex sulphide ores. Both operations employ conventional electrolytic zinc technology, and both precipitate the large amounts of associated iron as jarosite. Although the jarosite process works quite well, there is a general concern about the relative size and cost of the jarosite circuits, and at least one of the companies has examined an alternative to the jarosite process. Both companies are aware that metal

values, notably Ag and Zn, are being lost with the jarosite residue, and a number of studies have been carried out to try to minimize such losses. Lastly there is an underlying concern about the long term environmental acceptability of jarosite residue ponds with the problem being perhaps more acute at CEZ because it is situated in a built-up area.

Pressure leaching technology has been developed by Sherritt Gordon Mines Limited, and has been applied to conventional zinc concentrates at Cominco's zinc plant in Trail, B.C. and to high-iron feeds at Kidd Creek's zinc plant in Timmins, Ontario. The process has the dual advantage of forming elemental sulphur and of avoiding pyrite dissolution, thereby seeking to minimize the iron control problem early in the flowsheet. Despite the general lack of pyrite attack, however, there is still iron dissolution from the sphalerite and other iron-bearing minerals. Much of this dissolved iron precipitates in the autoclave as a jarosite-type compound which absorbs some of the silver and lead values. Sherritt is currently doing research on the control of losses into the jarosite and on the application of the technology to a number of different feeds including pyritic bulk concentrates. This is a promising technology which, if a few problems can be resolved, could eventually have important consequences for complex sulphide ore processing.

The New Brunswick Research and Productivity Council has developed a novel sulphation roast-leach process for pyritic bulk concentrates. In this process, ferrites invariably formed during roasting are subjected to hot acid leaching and the resulting concentrated iron solution is injected into the fluid bed roaster thereby avoiding the entire iron precipitation and filtration process. This technology has already been extensively tested in a small pilot unit, and a large pilot plant is about to be

commissioned. The novel iron control strategy adopted in this process could have applications elsewhere for iron control, and the RPC pilot campaigns will surely be closely watched by everyone involved with complex sulphide ores.

CANMET currently has a number of projects underway that are related directly or indirectly to the iron control problem in complex sulphide ores, and these activities are summarized in Figure 2. Chloride leaching processes are being developed for pyritic Zn-Pb-Cu bulk concentrates, and this technology has the advantage of largely avoiding pyrite dissolution, thereby seeking to control the iron problem early in the process and to avoid the precipitation of overwhelming amounts of this element. All work to date has been done on a bench scale, but plans for an integrated pilot plant are being developed in conjunction with Bacon, Donaldson Associates of Vancouver. Although this type of technology holds much promise, many inherent problems still have to be addressed, and the commercial realization of this type of leaching process may be further away than either the pressure leach or sulphation roast-leach technologies. Work is underway on the solvent extraction of iron from chloride leach solutions and on the extraction of various metals away from iron in both sulphate and chloride liquors, and such studies are of direct relevance to several iron control flowsheets developed for the treatment of complex sulphide ores. The regeneration of ferrous chloride leach solutions by chlorine gas or oxygen is also being examined, and such studies play an indirect role in the overall iron control strategy. A two-stage chlorination reactor has been built that allows the rapid regeneration of spent iron chloride leach liquors with a high chlorine utilization efficiency.

The last major area of CANMET activity in the iron control field centers on iron precipitation as jarosite-type compounds, and work is currently being channelled into three general areas.

One of the major problems associated with jarosites is their affinity for silver. It turns out that silver jarosite is very stable and it is nearly impossible to avoid the formation of silver jarosite or silver-bearing alkali or lead jarosites during the processing of Zn-Pb-Cu-Ag concentrates in sulphate media. By contrast, silver is strongly complexed in chloride media, and does not form a jarosite-type compound in concentrated chloride solutions. This may be a processing advantage for the chloride route although there are certainly other silver problems associated with chloride solutions. Several papers have already been published on the behaviour of silver in jarosite circuits, and work continues in this area.

It has long been known that potassium jarosite formation has several advantages for iron control, but the high cost of potassium salts has so far prevented the widespread use of this jarosite. Studies are currently underway, however, to recover alkalis from jarosite and to convert the iron to hematite. If such research efforts prove commercially successful, then the use of K-jarosite becomes feasible since the alkali would be largely recycled within the flowsheet. Accordingly, a thorough study of the factors affecting K-jarosite formation in both sulphate and chloride media has been carried out. The major difference between K-jarosite and the other alkali jarosite species is the significantly greater stability of the potassium member, and this stability has three major implications for the processing of complex sulphide ores. Firstly, the extent of iron precipitation increases, for a given set of conditions, when K-jarosite is used, and this is clearly desirable in plant practice. Secondly,

potassium jarosite can be formed over a broader range of concentration, pH and temperature, and this is generally desirable, but could lead to unwanted K-jarosite precipitation at low temperatures in purification units or in filter fabrics. Thirdly, K-jarosite incorporates higher levels of impurities, and this can be either desirable or undesirable depending on the value of the impurity.

The last area of current activity in the jarsosite field centers on the behaviour of arsenic in jarosite precipitation circuits. Arsenic is a common impurity in complex sulphide ores, and it is often controlled in the iron precipitation stage. Figure 3 illustrates the composition of the sodium jarosite precipitated at 97°C from solutions containing 0.2 M Fe^{3+} and various amounts of As^{5+}, added as sodium arsenate. It is evident that even modest amounts of arsenic in solution result in a significant arsenate content in the jarosite precipitate, and that the amount of arsenate in the precipitate increases systematically as the amount of dissolved arsenic increases. For the higher arsenic levels, over 90% of the initial As^{5+} in solution has been precipitated. Furthermore, as the arsenate content increases, the sulphate content decreases proportionally, apparently suggesting a substitution of AsO_4 for SO_4. The slight decline in the iron and sodium contents are also consistent with arsenate substitution for sulphate although the decline in sodium is more than anticipated. X-ray diffraction analysis of the various products indicated only sodium jarosite.

Although the data of Figure 3 seem to indicate that arsenate replaces sulphate, it is felt that such substitution does not, in fact, occur. The various X-ray powder diffraction patterns were indexed, and the resulting parameters are given in Figure 4 which illustrates the variation of the a and c parameters as a function of the mole fraction of arsenate in the

precipitate. It is clear that there is no systematic variation in either a or c with increasing arsenate content of the precipitate. Given that the arsenate anion is considerably larger than sulphate, this suggests that the arsenate is not actually substituted in the jarosite, but is present as an amorphous phase. To test this hypothesis, the precipitation of sodium jarosite was carried out at various temperatures and in the presence of 5 g/L As^{5+}, added as sodium arsenate. The resulting precipitates were all examined by X-ray diffraction analysis. At 100°C only sodium jarosite was evident in the X-ray pattern and this was true whether Debye-Scherrer or Guinier cameras were used. When the synthesis was repeated at 120°C, a faint trace of scorodite was evident in addition to the sodium jarosite pattern. At 140°C or at higher temperatures, major, well crystallized scorodite was evident in addition to the jarosite. The X-ray results suggest that although arsenate is being precipitated with the jarosite, it is not being structurally incorporated in the jarosite. This observation has important ramifications for the disposal of arsenic-bearing jarosite residues. Because the arsenic is present as a separate phase, it will leach under different conditions than the jarosite. If the residue pond is made slightly acidic to stabilize the jarosite, then the arsenic will leach; if the pond is made slightly basic for arsenic control, the jarosite will be decomposed. The presence of arsenic as a discrete amorphous phase greatly complicates the maintenance of the residue pond, and a similar argument could apply for other impurity elements as well.

Essential Iron		Associated Iron	
Zn-Pb-Cu Ores		Zn-Pb-Cu Ores	
$(Zn,Fe)S$	0-20% Fe	FeS_2	~46% Fe
$CuFeS_2$	30% Fe		
$(CuAgFe)_{12}Sb_4S_{13}$	0-10% Fe		

Fig. 1 - The two categories of iron contained in complex sulphide ores.

CANMET STUDIES ON IRON CONTROL

1. Chloride Leaching Processes for Bulk Concentrates
2. Solvent Extraction of Iron
3. Oxidation of Ferrous Chloride with Chlorine or Oxygen
4. Jarosite Precipitation
   - Ag Losses in Jarosite
   - K-Jarosite Formation
   - As Incorporation in Jarosite

Fig. 2 - Summary of current CANMET activities related to the iron control problem in complex sulphide ore processing.

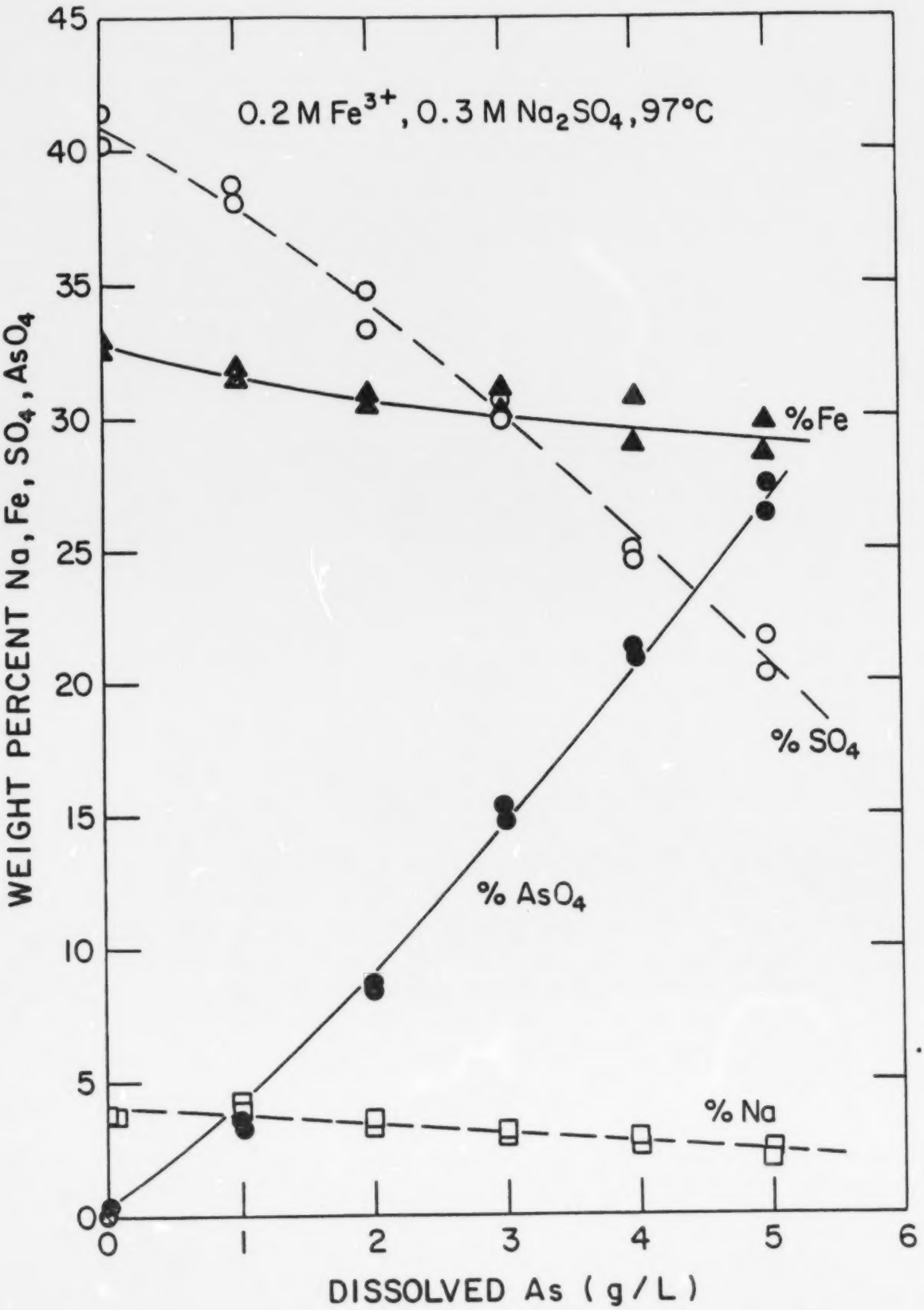


Fig. 3 - Composition of the sodium jarosite precipitate formed in the presence of As^{5+}.

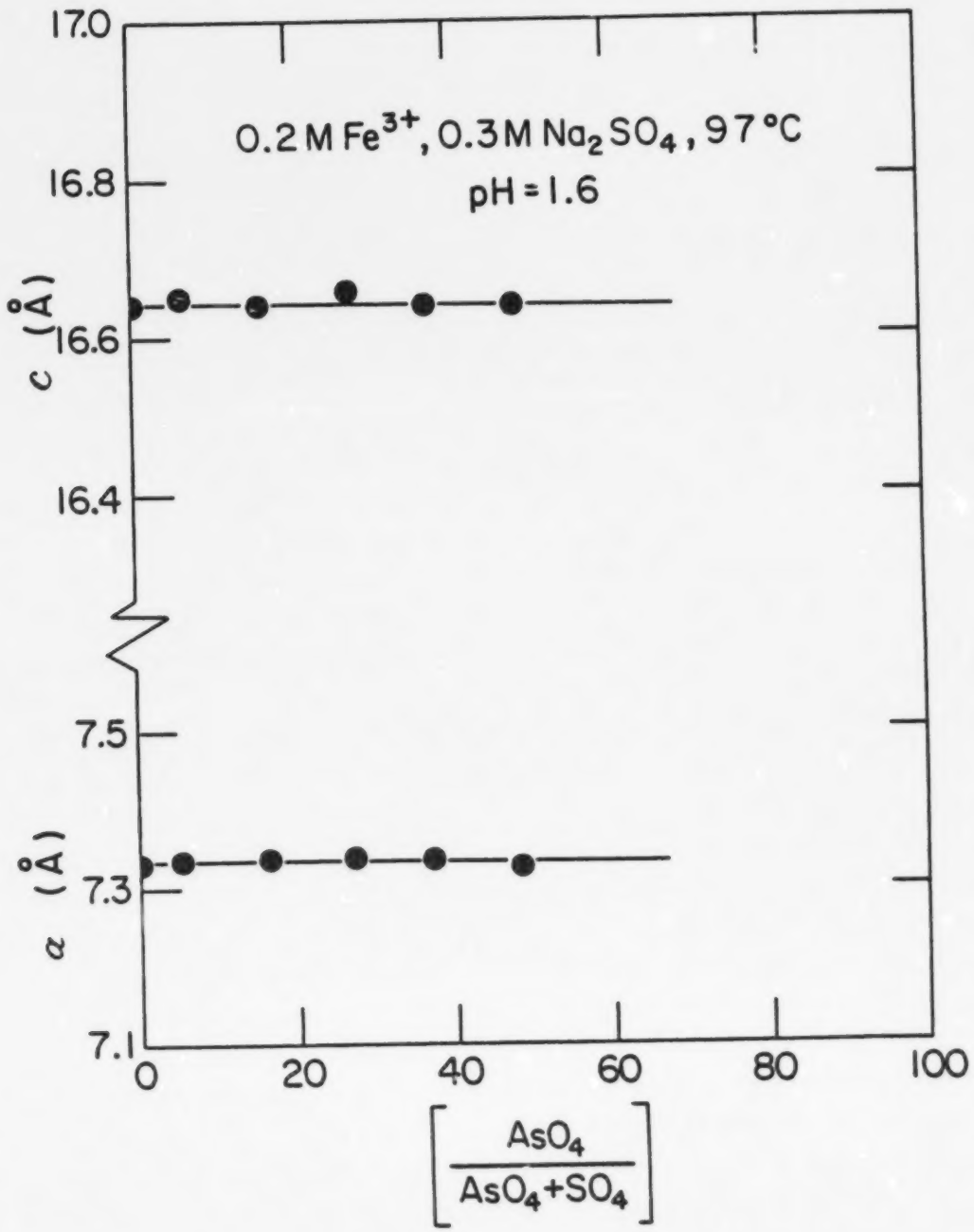


Fig. 4 - Variation of the a and c parameters of the sodium jarosite with increasing mole fraction of arsenate in the precipitate.

CEC/CANMET MEETING OTTAWA MAY 13/14 1985

IRON REMOVAL FROM COMPLEX ORE LEACH LIQUORS BY HYDROLYTIC STRIPPING

Contract No. MSM 094 UK
Project Leader: Dr. A.J. Monhemius
Dept. Metallurgy & Materials Science,
Imperial College,
London SW7 2BP, UK.

The reaction of iron-loaded Versatic acid with water at elevated temperatures to produce iron oxide with the simultaneous regeneration of the free acid has been studied:

$$2FeV_{3(org)} + 3H_2O \rightarrow Fe_2O_3 + 6HV_{(org)}$$

Experiments have been carried out in a batch-stirred autoclave at temperatures of 150 and 185°C. On-line pressure filtration has been used to recover the precipitated iron oxide from the reacted organic phase. It has been found that reproducibility of slurry filtration rates, which are indicative of the physical characteristics of the precipitated iron oxide particles, has been very difficult to obtain in the batch experiments. The solids have been characterised by a number of techniques including XRD, DTA, TGA, electron microscopy and laser particle size analysis.

Hydrolytic stripping at 150°C produced hematite, Fe_2O_3 only, but the reaction time of 30 min. to precipitate a solution initially containing 15g Fe^{3+}/L was considered to be too long for industrial application. The reaction time can be reduced to 10 minutes by operating at 185°C, but the oxide product is then a mixture of hematite with magnetite and/or goethite. Apparently similar experimental conditions could give rise to slurries with greatly differing filtration rates; particle size analysis showed some correlation between slow filtration rates and the presence of sub-micron particles in the precipitates. However, electron microscopy showed that particle shape, as well as size, affected filtration rates. The filter cakes, produced by pressure filtration, contained 15-20 wt % organic phase. It was found that the organic could be efficiently recovered by vacuum-drying the cake, to give an iron oxide product containing about 1 wt % carbon.

It was concluded that reproducibility would only be achieved by running continuous precipitation tests rather than batch experiments. Modifications have been made to the autoclave to enable it to be run in continuous mode, but no continuous hydrolytic stripping experiments have yet been carried out.

The analogous process, called precipitation stripping, in which iron is precipitated from Versatic acid at 100°C and atmospheric pressure by reaction with dilute aqueous acids, has also been studied in some detail. This process can be considered to occur in two stages:

Stripping $FeV_{3(org)} + 3H^{+}_{(aq)} \rightarrow Fe^{3+}_{(aq)} + 3HV_{(org)}$

Precipitation $Fe^{3+}_{(aq)} + 2H_2O_{(aq)} \rightarrow FeOOH_{(S)} + 3H^{+}_{(aq)}$

Acid in the aqueous phase to give a pH less than about 3 is necessary to initiate the stripping reaction. Provided that the rates of stripping and precipitation are made equal, the system is self-buffering and the stripping and precipitation of iron can proceed to completion.

Initially sulphuric acid was used in the aqueous phase. Batch reactions were successfully carried out, the iron precipitating as goethite. Continuous experiments however were less successful since it was found that the iron products contained considerable amounts of sulphate due to the formation of basic sulphates.

In order to avoid contamination of the iron product, precipitation stripping using aqueous soluble organic acids was investigated. Formic acid was found to be excellent - the aqueous phase was self-buffering at about pH2; no pH control was required. Furthermore the iron precipitated from solution as sub micron hematite - an ideal pigment material. Using 0.4M formic acid at 100°C, Versatic acid containing 22g/L Fe^{3+} can be completely precipitated in an hour. The phases are easily separated by centrifuging and the solids are aqueous wetted. Many variables have been investigated in the formic acid stripping system and the system has been found to perform extremely well. Recently continuous 4-stage precipitation stripping tests have been carried out. Increased residence times compared with batch reactions are necessary to achieve low iron levels in the organic phase but otherwise the continuous system has been found to operate satisfactorily.

RENCONTRE C.E.E. - CANMET

POLITIQUE INDUSTRIELLE DE RECHERCHE DANS LE DOMAINE DE L'ÉLIMINATION DU FER À MINEMET RECHERCHE

François RICALENS

Dans le Groupe IMETAL, comme dans toutes les sociétés productrices de métaux non-ferreux, la purge du fer est un problème fondamental pour lequel il n'existe pour l'instant aucune solution complètement satisifaisante tant au plan économique qu'au plan de l'environnement.

Pour mémoire, en ce qui concerne les minerais de nickel de Nouvelle-Calédonie, le problème ne se pose pas car le fer réduit dans les fours électriques est vendu dans le ferro-nickel, cependant que le fer encore présent dans la scorie est suffisamment réfractorisé pour éviter toute atteinte à l'environnement. Le problème de la purge du fer n'existerait qu'en cas d'utilisation d'un procédé hydrométallurgique.

Cas des résidus d'usines à zinc

A l'heure actuelle, les résidus d'usines à zinc produits dans l'usine de PERTUSOLA SUD à Crotone sont soumis à une réfractorisation dans un cubilot qui permet en outre de récupérer le zinc, le plomb, l'argent et les métaux présentants un sous-oxyde volatil et qui se trouvent dans le résidu.

La scorie est suffisamment réfractorisé pour qu'aucune atteinte à l'environnement ne soit portée. La scorie pourrait même être un facteur de santé dans la mesure où elle pourrait être utiliser pour des opérations de sablage et éviterait l'utilisation de ces composés qui peuvent provoquer une malalie fort grave : la silicose.

Cette solution, qui permet d'éviter tout résidu des usines à zinc, nous semble particulièrement satisfaisante. Toutefois, il apparaît de plus en plus que le cubilot n'est probablement pas l'outil métallurgique le plus économique dans les conditions actuelles pour obtenir la valorisation des différents métaux que nous avons cité précédemment et la production d'une scorie utilisable pour certains usages évitant tout stockage de résidus.

MINEMET RECHERCHE, avec l'aide de la C.E.E., est engagée dans un travail de réflexion sur les différentes options offertes pour remplacer ou améliorer le cubilot en réduisant la consommation la plus coûteuse, à savoir celle du coke qui sert, dans cet outil, de réducteur et de producteur de chaleur.

Cas des nouveaux procédés en voie chlorure

La purge des procédés en voie chlorure développée par MINEMET RECHERCHE est un des problèmes-clés de ces études dès lors que la chalcopyrite doit être traitée.

De nombreuses voies ont été proposées mais la voie choisie par MINEMET RECHERCHE est particulièrement intéressante car elle est peu coûteuse et elle conduit à des résidus peu polluants. Toutefois, un certain nombre de problèmes restent à résoudre si l'on désire valoriser un jour le fer ainsi précipité.

Rappelons le principe de la technique développée par MINEMET RECHERCHE. Cette technique consiste à précipiter un hydroxy-oxyde que l'on désigne sous le nom de "goethite" par oxydation du fer ferreux en fer ferrique dans des domaines ou il ne peut pas exister beaucoup de fer ferrique en solution. Deux manières existent de réduire le domaine d'existence du fer ferrique en solution : jouer sur le potentiel redox et jouer sur le pH. Dans le cas qui nous occupe, compte tenu de l'existence en solution du cuivre, c'est en jouant sur le potentiel redox que l'on réduit le domaine d'existence du fer ferrique en maintenant une teneur élevée en ions cuivreux. En travaillant ainsi, les teneurs de la goethite en cuivre et en chlore sont relativement élevées.

Une des études les plus intéressantes qui ont pu être menées a démontré qu'il était possible de réduire quasiment à zéro la teneur en cuivre et en métaux lourds de la goethite en choisissant soigneusement un certain nombre de conditions opératoires. En revanche, la question de la teneur en ions chlorure, bien que partiellement résolue corrélativement à la diminution en cuivre, reste toutefois posée.

Certains éléments d'études qui ont été faites montrent que cet objectif n'est pas inaccessible mais les paramètres permettant de réduire à un niveau très faible les ions chlorure ne sont pas complètement maîtrisés et doivent faire l'objet d'une étude beaucoup plus approfondie.

L'AIR LIQUIDE - SASSENAGE -

DIRECTION DES CENTRES DE RECHERCHE ET DE DEVELOPPEMENT
CENTRE DE RECHERCHE ET DE DEVELOPPEMENT DE GRENOBLE

SERVICE GENIE CHIMIQUE

JPB/mpc - 85. 7076 -

ETUDE DE LA PRECIPITATION DU FER PAR L'OXYGENE GAZEUX DES SOLUTIONS ACIDES CHLORUREES DE LIXIVIATION PAR LE CHLORURE CUIVRIQUE DES MINERAIS SULFURES COMPLEXES

Projet C.E.E. N° M.S.M. 049 F(SD)

REUNION C.E.E./CANMET - OTTAWA (Canada) 13/14 Mai 1985

Jean-Pierre BURZYNSKI

SASSENAGE, le 10 Mai 1985

SERVICE GENIE CHIMIQUE
85. 7076

1. CADRE DE L'ETUDE

Ce travail s'inscrit dans le cadre du traitement des minerais sulfurés complexes - mélange de chalcopyrites CuFeS2 - galène PbS - blende ZnS dans une gangue constituée principalement de pyrite FeS2 ou de pyrrhotine $Fe_{1-x}S$. Le traitement physique de ces minerais conduit à un concentré global qui sera l'alimentation d'un procédé métallurgique.

Le procédé développé par la Société MINEMET - RECHERCHES (Figure 1) est dans son principe une mise en solution de tous les minéraux sulfurés contenant des métaux non ferreux (donc sélective vis-à-vis de la pyrite) par une solution de chlorure cuivrique (oxydante grâce au couple Cu^{++}/Cu^{+}) dans une solution fortement concentrée en chlorure de sodium , à une température comprise entre 80 et 105°C. Ce procédé comporte ensuite les étapes suivantes :

- précipitation du fer,
- extraction du cuivre par un solvant organique spécifique,
- extraction du zinc par un solvant organique sensible au fer.

L'étape la plus délicate de ce procédé concerne la précipitation du fer sous forme de goethite en même temps que la régénération du chlorure cuivrique employé à l'attaque.

La méthode préconisée et utilisée par L'AIR LIQUIDE pour cette déferrisation consiste en une oxydation à l'oxygène pur sous pression et à une température comprise entre 80 et 150°C. Les avantages de ce procédé sont liés essentiellement à l'utilisation de l'oxygène pur comme agent d'oxydation.

./.

Ces avantages sont les suivants :

- la cinétique d'oxydation est augmentée et contrôlée permettant par là même d'ajuster la vitesse de précipitation de façon à obtenir un précipité de bonne qualité,
- au niveau énergétique, on supprime les pertes thermiques dues à l'entraînement de vapeur d'eau durant les purges du ballast d'azote,
- au point de vue technologique, la pression et le volume du gaz à introduire dans le réacteur étant plus faibles, son volume est réduit et sa construction est simplifiée.

./.

2. OBJECTIF DE L'ETUDE

On peut distinguer trois phases dans cette étude :

- une première phase consiste en la détermination de la loi cinétique d'oxydation du Fe(II) et du Cu(I) par l'oxygène gazeux dans les conditions du procédé MINEMET.
- la seconde consiste en l'étude de la précipitation de l'hydroxyde ferrique : on détermine l'influence des principaux paramètres de contrôle (température, pH, pression partielle d'02) sur l'efficacité de cette précipitation. Il conviendra en particulier de minimiser l'entraînement du cuivre et des chlorures dans le précipité.
- la troisième phase consiste en la définition des principaux paramètres opératoires permettant le fonctionnement optimal d'un réacteur pilote d'oxydation capable de traiter quelques m³/h de solution en un temps de séjour d'environ une heure.

Ce travail est à rapprocher de l'étude entreprise lors du précédent contrat (contrat C.E.E. n° 018 MPPF). Il avait été alors étudié la cinétique de l'oxydation et la précipitation de l'hydroxyde FeOOH dans le cas où la lixiviation se fait en milieu (FeCl3 - HCl) riche en fer ferrique.
La présente étude est une extension dans un domaine du rapport des concentrations $[Fe]_T / [Cu]_T = 0,2$ inversé par rapport au premier contrat (= 5) et pour des teneurs en chlorures plus importantes.

./.

Nous avons étudié l'influence du pH, de la température et de la pression partielle d'oxygène sur les cinétiques d'oxydation du Cu(I) et du Fe(II). Pour cela, nous avons utilisé l'installation expérimentale de la figure 2 : un réacteur agité en verre permet de travailler sur un volume de 1 litre de solution MINEMET. L'oxydation se fait en batch avec des prélèvements à des temps donnés pour la détermination des vitesses de réaction ; la consommation d'oxygène peut également être suivie en fonction du temps. Température et pH sont suivis et contrôlés en permanence dans le réacteur. Les analyses des précipités sont effectuées par la Société MINEMET - RECHERCHES.

./.

3. CINETIQUES D'OXYDATION

On a trouvé qu'il fallait prendre en compte les réactions suivantes :

$$Cu(I) + 1/4\ O2 + 1/2\ H2O \longrightarrow Cu(II) + OH^-$$

$$Cu(II) + Fe(II) + 3\ OH^- \longrightarrow Cu(I) + \underrightarrow{FeOOH} + H2O$$

La réaction d'oxydation du Fe(II) par le Cu(II) formé est rendue possible par l'élimination du Fe(III) qui précipite.

On a montré que la présence de fer ferreux accélère la cinétique de la réaction d'oxydation du Cu(I) : le transfert global de l'oxygène est alors accéléré d'un facteur variant entre 1,5 et 3 (Tableau 3, Figure 3) et s'écrit :

$$R02 = E \cdot k_La \cdot [O2]_i$$

où :

R02 : mole/l/s

k_La : coefficient global de transfert en $sec.^{-1}$

$[O2]_i$: concentration de l'oxygène à l'interface en mole/l

En l'absence de fer ferreux, la vitesse d'oxydation du Cu(I) est une constante qui, pour les conditions standard, se stabilise entre 1,3 et 2.10^{-4} mole/l/s. Dans ces conditions, le transfert global de l'oxygène s'exprime par la relation :

$$R02 = k_La \cdot [O2]_i$$

dans la mesure où le taux de conversion est inférieur à 80%. Au delà de cette valeur, la cinétique d'oxydation du Cu(I) devient suffisamment lente pour que la concentration en oxygène dissous dans la masse liquide ne puisse plus être supposée nulle : on se trouve alors dans un régime intermédiaire d'oxydation.

./.

On a observé que la vitesse d'oxydation du Cu(I), contrôlée par le transfert de l'oxygène, dépend peu du pH et de la température.

On a également fait varier la pression partielle d'oxygène entre 0,3 et 1 bar. La vitesse d'oxydation du Cu(I) varie de façon linéaire en fonction de ce paramètre (figure 4).

Nous poursuivrons ce travail au cours du 4ème semestre en modélisant le fonctionnement du réacteur pilote continu à l'oxygène ; cette étude sera incluse dans le rapport final d'étude.

Jean-Pierre BURZYNSKI

SERVICE GENIE CHIMIQUE
85. 7076

CARACTERISTIQUES DES SOLUTIONS ISSUES DE LA LIXIVIATION :

NaCl	: 250 g/l	Fe	: 4,4 g/l
Pb	: 7,6 g/l	Cu^T	: 49 g/l
Zn	: 16,3 g/l	Cu(I)	: 19 g/l

CONDITIONS DES ESSAIS :

TEMPERATURE VARIABLE	: 80<T(°C)<110
PRESSION PARTIELLE C2	: 1 BAR
Cu (I) INITIAL/Cu(I) FINAL	: 5
VOLUME LIQUIDE	: 1 litre
VITESSE DE ROTATION	: 1300 T/min.
AIRE INTERFACIALE	: 3,5 CM^{-1}
DEBIT GAZEUX	: 50 l/h
ARBRE CREUX	
pH variable	: 1 < pH < 3

CONDITIONS EXPERIMENTALES

T A B L E A U

SERVICE GENIE CHIMIQUE
85. 7076

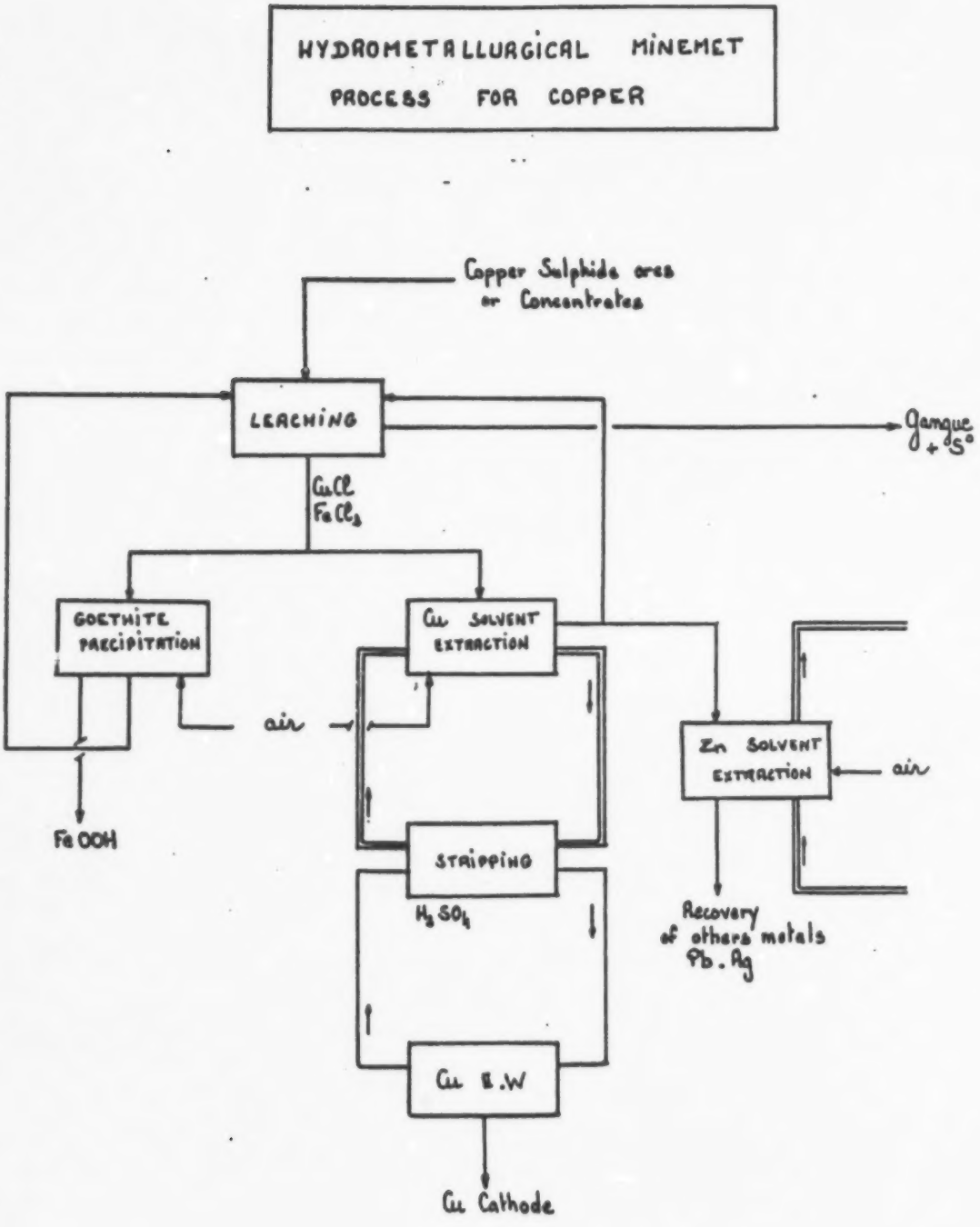
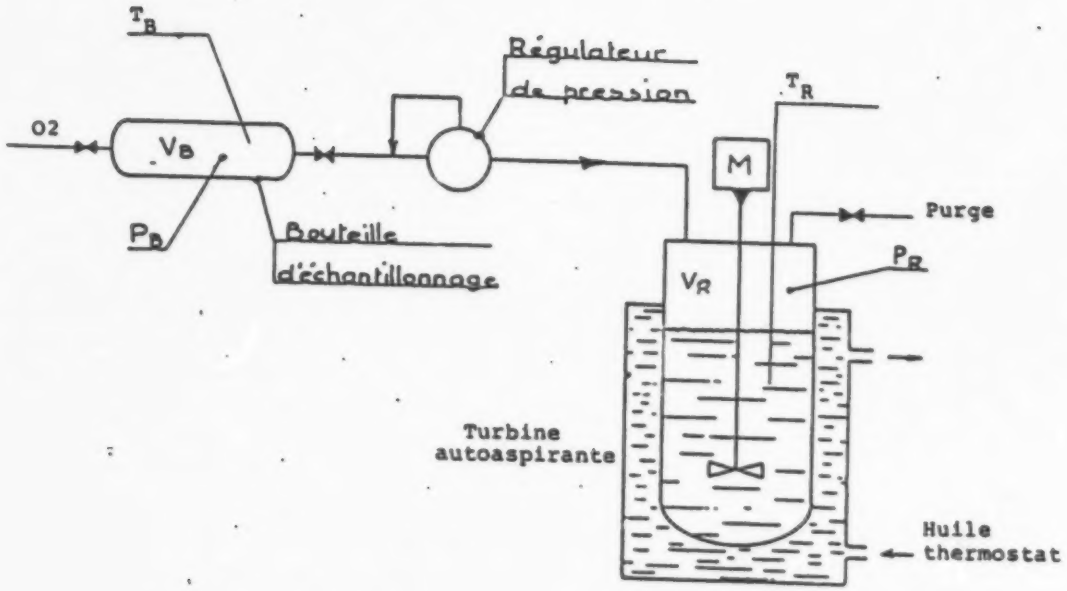


FIGURE 1

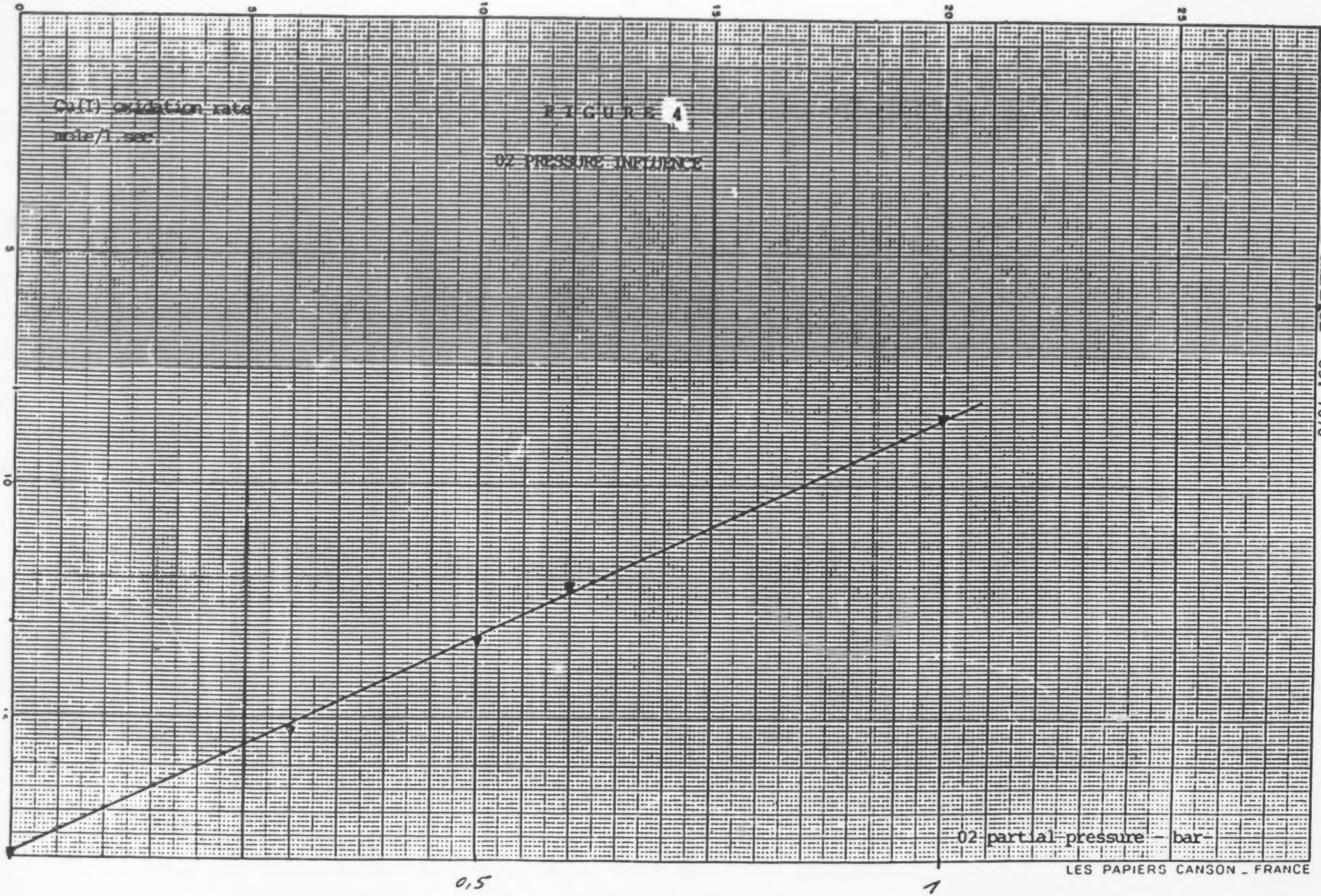
SERVICE GENIE CHIMIQUE
85. 7076



V_B=0,3251
V_R=0,75 l
R=0,082 bar.l/°K/mole

SCHEMA DE PRINCIPE DE L'APPAREILLAGE UTILISE

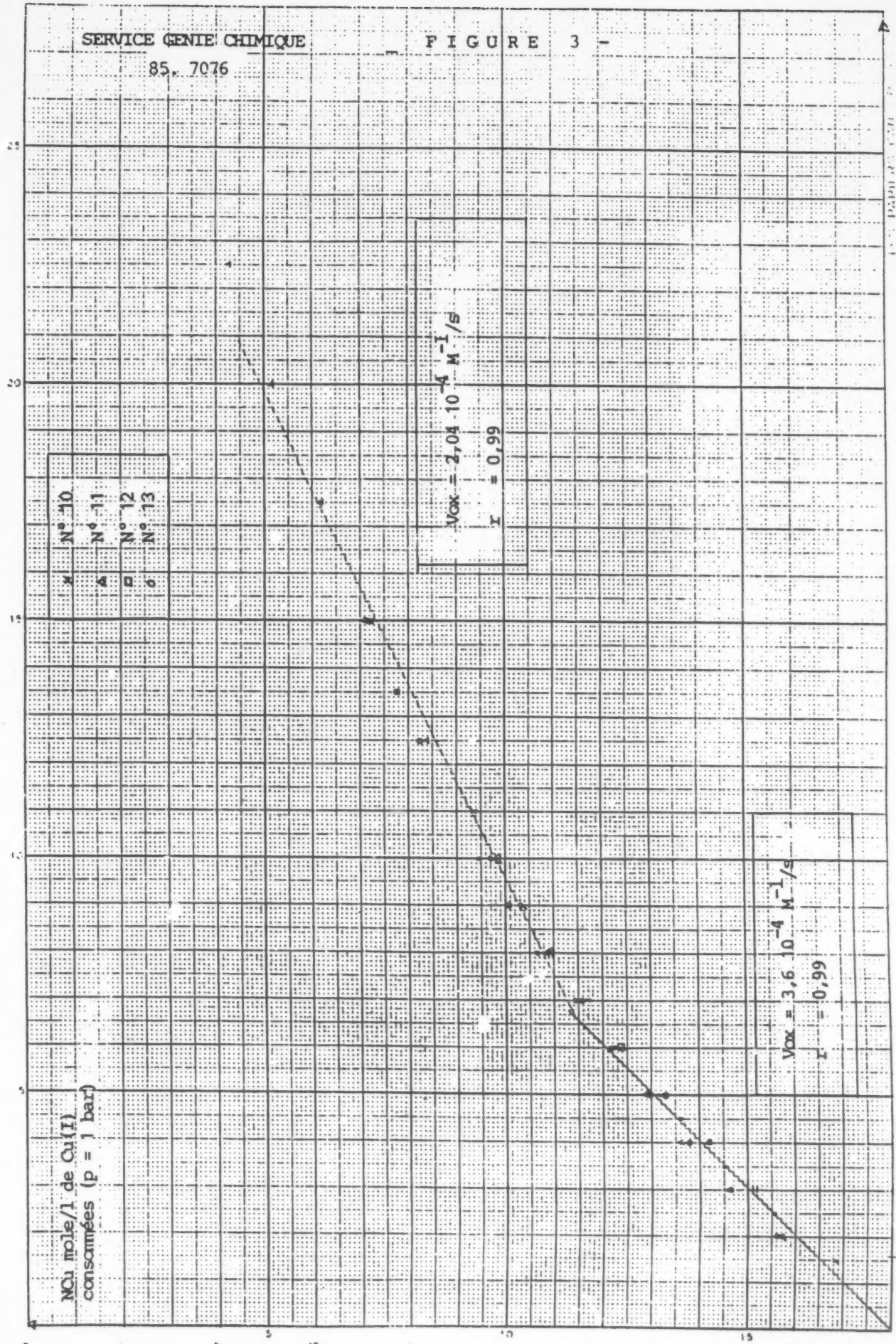
FIGURE 2


0
5
10
15
20
25
Cat1) oxidation rate
mole/l.sec.
FIGURE 4
02 PRESSURE INFLUENCE
02 partial pressure - bar -
0,5
1
SERVICE GENIE CHIMIQUE - 85. 7076
LES PAPIERS CANSON - FRANCE

SERVICE GENIE CHIMIQUE

FIGURE 3 -

85. 7076



Annex II - Technical Research Proposals (EEC/CANMET)

Liberation Process Modelling

The desire and scope for collaboration with EEC contractors on this topic is very strong. In process modelling many parameters are needed including liberation, but skepticism on liberation measurements still exist. Therefore two topics must be examined:

(I) Liberation: It is proposed to prepare two types of known standard samples (a) Two phase samples that can be characterized;
(b) Multiphase samples that would be accurately analysed.

Standard polished sections would be prepared from several ores, and these along with the powders would be circulated to the laboratories of EEC and Canada analysis. The analytical reports from each laboratory would give full analytical condition.

The co-operative work is proposed as follows:

(a) Preparation of two phase samples would be the responsibility of G. Barbery, Laval University, Canada, and M.P. Jones, Imperial College (EEC).

(b) Preparation of multiphase samples would be the responsibility of W. Petruk, CANMET, D. Hendricks, Brunswick Mining and Smelting (Canada) and Mr. A. Broussaud, BRGM (EEC).

(II) Process Modelling (Preliminary Proposal) A unit process model that covers the entire circuit should be developed on a co-operative basis between the EEC workers and Canadian workers with the Canadian contribution co-ordinated by CANMET. The model should have a large input from industry, with fundamental and laboratory input from universities. There would be an exchange of plant data between the researchers covered by EEC contracts and the Canadian workers, and a joint evalution of the data, and in developing the resulting software.

It is proposed that, from the Canadian side, the data would be collected on material in the mill of Brunswick Mining and Smelting, and it would be augmented by studies at Laval University and University of British Columbia. The work and results would be co-ordinated in Canada by CANMET, and would be submitted to the concerned European EEC contractors for co-operative input and interpretation.

An outline of the project is:

A. Goal
   - develop process model
B. Lab Experiments
C. Plant experiments
D. Model building
E. Test model in plant and simulator

Involvement

CANMET { BMS, Laval, U. B.C.

EEC

An initial meeting for the purpose of fully characterize the project should be arranged as soon as possible, and to be followed by periodical meetings. A minimum of 3 days are envisaged to permit discussion on the progress of the project.

Modelling of Liberation and Mineral Separation Procesing
(Preliminary Proposal)

The Contractor (Imperial College) will colaborate with CANMET, Brunswick Mining & Smelting, Laval University, University of British Columbia, B.R.G.M. and tne Warren Spring Laboratory in the development of liberation models and models for flotation and for gravity separation circuits.

M.P. JONES

Technical Information

In addition, the measuring methods that will be used for characterising the detailed mineralogy of all specimens must be checkd against carefully-prepared standard materials. The production of these standards will be costly and time-consuming and the costs and the effort should be shared among the collaborators. Furthermore, selected specimens (other than standards) that have been measured by one party should be made available so that they can be independently checked - in particular, this will apply to materials that only contain trace amounts of rare phases and/or significant proportions of very fine-grained materials.

Comparisons will be made between the results obtained by one-dimensional and by two-dimensional measuring techniques (i.e. results produced in Europe and in Canada, respectively).

Further preliminary details given in submission by Dr. Petruck, CANMET.

Liberation Modelling (Draft Provisoire)

BRGM
A. BROUSSAUD

Informations techniques

1. Libération minérale

   a) Sélection des cibles et préparation d'échantillons

      - un minerai simple (fer du Canada ?) pour lequel l'analyse densimétrique permettra de déterminer la libération réelle à 3 dimensions

      - un minerai sulfuré complexe fin

   b) Echange d'échantillons et comparaison de résultats avec différents laboratoires canadiens et européens - conclusions sur la précision des méthodes de préparation d'échantillons, de mesure à 1 dimension et à 2 dimensions, et de reconstruction à 3 dimensions - recommendations pour le traitement du minerai sulfuré complexe.

2. Modélisation du procédé

2.1. Amélioration des méthodes de Conception Assistée par Ordinateur en minéralurgie

   - inventaire de modèles simples de concassage, broyage, flottation, filtration, séparation magnétique, gravimétrie...

   - incorporation de ces modèles dans les logiciels du BRGM

   - adaptation éventuelle de logiciels développés par ailleurs

   - test de ces modèles et logiciels avec des données industrielles : évaluation de leur validité pratique

2.2. Développement du contrôle d'usine par ordinateur en minéralurgie

   - sélection d'une cible industrielle (ou pilote)

   - mise en place d'un système d'acquisition de données en ligne, et éventuellement de boucles de régulation locales

   - développement de logiciels spécifiques pour fournir aux opérateurs de l'usine des informations en ligne sur :

     . le bilan matière
     . les réglages recommandés à partir de modèles de simulation et d'optimisation.

Remarque : Les points 1 et 2.1. devraient faire l'objet de coopération active avec des laboratoires canadiens et européens.

Use of Knowledge Based Systems in Conjunction with Process Simulation Techniques to Predict the Operational Performance of Ore Grinding Installations and Froth Flotation Plant

Warren Spring Laboratory, UK

G.W. CUTTING

Technical information

The simulation of grinding and froth flotation plant performance has reached a decision point, where the techniques advocated and used today do not supply many of the answers needed at the practical level; particularly regarding the metallurgical performance of grinding plant and the interactions with subsequent mineral processing steps. A division is noted between the use of relatively simple calculation methods in conjuntion with much professional judgement and expertise and more advanced numerical methods, which seek to maximise engineering design techniques. Grinding and flotation are complex and diversified fields and both of the above methods offer part of the overall solution needed.

This project aims to unite the wealth of judgements, expertise, rules of thumb etc available generally with proven numerical methods to provide a simulation technique capable of supplying answers to a wide range of problems encountered practically in the design of grinding and flotation installations. The expertise etc will be structured into a knowledge based system (expert system) and this systems will be used to transform the expertise into parameter formats for subsequent use in the numerical algorithms of the overall simulation method. In practice a user will come to the system with a problem and some "hard" data in numerical form (laboratory testwork etc). The simulation system will assimulate the problem structure and given data and then enter the knowledge bank of the expert system and generate the parameter set for the solution algorithms. The user will be presented with a summary of the decision made by the expert system, and these can be either accepted or further searches for parameter sets can be made with the user supplying overriding factors etc.

The proposed system is split into two sections where the first section consists of the process models and the second section is the expert system consisting of a knowledge bank (compendium of rules and judgements etc) and an inference engine, which enters the knowledge bank, as instructed by the user, and makes decisions regarding the parameter sets to be used in the simulation. The knowledge bank is generated and updated from the accumulated process knowledge by a knowledge acquisition system.

The end result of the programme of work will be a simulation method which communicates with the user in practical terms and supplies answers to problems of practical interest. Such a system will be available for commercialisation, both as a subsection of a more general problem and for direct software sales.

The project work is scheduled around a three (or more) year period. The work will involve both the collection of process data and the development of computer based performance prediction techniques. The process data will consist of descriptions of the operational performance in metallurgical terms of a wide range of grinding and flotation installations operating on various mineral ore types; together with the local expertise, rules of thumb, etc at each of the installations. The collected "knowledge" from the plants will be combined with similar knowledge available at WSL and used to construct the expert systems, which will provide the parameters set for the numerical performance prediction algorithms. Maximum emphasis will be placed upon expert system development and, as far as possible, existing numerical techniques will be used; however, some development of the numerical systems will be necessary to ensure an efficient match between the knowledge based and numerical systems. A protoype system will be developed at the halfway stage in the project plan and this system will be used, in conjunction with invited users from industry, to both verify that the existing developoments are producing systems of use to industry generally and to provide pointers and guidelines for the development work in the second half of the project.

Liberation Modelling

UNIVERSITE LIBRE DE BRUXELLES
G. PANOU

Informations techniques

Notre programme pour 2 ou 3 ans à partir de 1986 fait suite à nos travaux actuels et comprend la recherche des possibilités de traitement sur micro ordinateur de problèmes précis de contrôle et de prévisions à partir de données courantes dans les laveries et d'un minimum, à préciser. de travaux expérimentaux de durée : la durée la plus courte possible. Des vérifications seront prévues pour tester la fiabilité des ré

Une attention particulière sera portée à l'adaptation de nos modèles et logiciels de façon à établir une liaison entre broyage et enrichissement. Il s'agira de répondre à la question suivante : étant donné, dans un cas précis, une série de broyages, conduisant chacun à une distribution granulométrique et à une libération particulière, quels sont les rhéogrammes optimums correspondant à chaque cas et, en précisant les critères (variables selon le cas), quelle est la meilleure combinaison.
Dans le modèle de broyage, la distinction entre fonctions de sélection et de broyage sera maintenue de façon à utiliser la première comme moyen de contrôle de l'efficacité du broyeur.

En outre, nous comptons informatiser et étendre la technique géostatistique mise au point, pour des schiste bitumineux, de recherches d'"entités", permettant une préconcentration poussée de grains très fins, après un broyage grossier; deux nouvelles applications prévues, en Belgique, pour extraire des minéraux non métalliques d'une diorite et, au Zaïre, pour le traitement d'une pegmatite pauvre à grains très fins.

Ces travaux nécessiteront probablement aussi la modification de nos modèles actuels de concentration, fondés, pour les séparations gravimétriques et magnétiques, sur les courbes de lavabilité, et pour la flottation sur l'aptitude de grains à la flottation.

Annex II - Technical Research Proposals (EEC/CANMET)

Fine Particle Technology

The work on fine particle technology under the present contract period would touch on three major topics which are complementary and of interest to all parties because of their direct application to industry. The first one, to be done by Warren Spring Laboratory, will consider the development of a model to predict the interaction between fine minerals and reagents to help in the. prediction of the behaviour of the complex syste- development of new selective reagent systems.

The second one, to be done by Minemet Recherche, would concentrate on the recovery of tetrahedrite. Silver losses are a problem affecting all fine grained complex sulphide ores products. The work would cover the fundamentals of the mineral and its interaction with chemical and flotation parameters. It would include two ores of their choices plus Canadian samples.

The third topic, to be done by CANMET, include the beneficiation of cassiterite which is present in most complex, fine grained ores which are of interest to the cooperating parties. In depth mineralogy will determine mode of occurency and properties necessary to ensur its separation from gangue minerals and techniques to either float the gangue or the tin minerals. Part of the work will include the evaluation of fine bubble produced in flotation machines.

Ores relevant to the study will be chosen.

Reagents in Fine Particle Processing

Warrens Spring Laboratory

P. PARSONAGE

Technical Information

The areas of work proposed are as follows :

Phase 1) Development of a theoretical model to predict the interaction between mineral sand reagents to help in the prediction of the behaviour of complex systems and the development of new selective reagent systems. Specifically the aims will be to predict

i) the state of mineral surfaces and the solution composition in mixed mineral pulps

ii) the concentration of ionic species formed when reagents are added

iii) the relationship between structure of reagents and the way they absorb at interfaces.

Techniques for characterizing suspensions will include : laser light Fraunhofer diffraction will be used for size distribution measurement; laser Doppler velocimetry for measurement of electrokinetic properties; photon correlation spectroscopy for measurement of reagent molecular weight of polymeric reagents.

Computer programs will be developed in connection with parts i), ii) and iii) above. A critical survey of the literature will also be made.

Phase 2) Application of results from Phase 1 will be applied to operating systems.

Phase 3) Selective magnetic coating process :

i) investigation of the surface chemical basis of the process

ii) extension of the process to a wider range of systems

iii) pilot plant testing of the process

iv) investigation of sources of magnetic coating material.

Etude des propriétés de flottation des cuivres gris afin d'améliorer la récupération de l'argent

MINEMET RECHERCHE
J.J. PREDALI

But de la recherche

Dans de nombreux minerais sulfurés complexes, l'argent est présent à des teneurs non négligeables (une à plusieurs onces par tonne) et sa récupération est un facteur économique important dans la rentabilité du projet. En général, les résultats industriels sont toutefois médiocres et la récupération totale ne dépasse pas 50 %. Ce résultat est dû à la minéralogie complexe des porteurs de l'argent qui peuvent être :

- la galène
- les cuivres gris (tétraédrite, ...)
- l'argentite ...

Les cuivres gris qui sont souvent le porteur principal, mais avec une phase en trace dans le minerai, ont des propriétés de flottabilité mal maîtrisées actuellement. Ainsi, les retrouve-t-on parfois avec le concentré de plomb ou celui de cuivre; alors l'argent est bien payé au mineur. On le retrouve aussi parfois dans le concentré de zinc ; alors l'argent est beaucoup moins bien payé au mineur et, le plus souvent il n'est pas payé, et, dans le pire des cas - malheureusement le plus fréquent - les cuivres gris ne flottent pas et sont perdus dans les stériles. Cette constatation a été faite pour de nombreux minerais complexes à maille de libération fine en Europe, au Canada, mais aussi pour des minerais plus classiques, notamment certains gisements du Pérou.

Le but de la recherche est de mieux connaître la flottabilité des cuivres gris afin de pouvoir les flotter dans les concentrés de cuivre ou de plomb ou dans un concentré spécifique afin que la récupération soit augmentée et que l'argent soit mieux payé.

Phases du projet

Cette recherche nécessite à la fois des travaux de base pour connaître les paramètres de flottation des cuivres gris - qui sont en fait un groupe représenté par plusieurs phases - et aussi des travaux d'adaptation de ces paramètres dans les différents cas des minerais connus. Il est donc prévu de mener ces deux activités en parallèle.

a) Sélection des cuivres gris à étudier

Il s'agit de déterminer quelles sont les phases les plus fréquentes dans les minerais étudiés : tétraédrite, ténnantite, ...

Il faudra ensuite se procurer des échantillons assez purs de ces phases pour les études de base.

b) Etude de base sur la flottabilité des cuivres gris seuls et en présence des autres sulfures : chalcopyrite, galène blende, pyrite.

Durée estimée de a) et b) : 18 mois

c) Application des propriété de flottabilité à différents minerais

Cette phase doit attendre que a) et b) soient suffisamment avancées pour démarrer. Elle comprendra alors les étapes suivantes :

- définition des résultats obtenus par les méthodes classiques pour la récupération de l'argent

- modification des méthodes classiques pour accroître la récupération des cuivres gris

Il est prévu de traiter en détail deux minerais et, si les résultats sont prometteurs, d'effectuer une étude préliminaire en laboratoire pour deux ou trois échantillons de minerais canadiens pour lesquels se pose ce problème : Kidd Creek Company, une usine de Cominco et une de Noranda. Bien entendu, il est prévu que ces mines fourniront, outre les échantillons, tous les résultats déjà disponibles pour accélérer le démarrage des travaux.

La durée totale de cette phase sera de 18 mois.

ANNEX II - Technical Research Proposals (EEC/CANMET)

Iron Control in Hydrometallurgy

Although certain differences exist in the economic and environmental milieu between Europe and Canada, it is felt that a use collaborative programme could be develop on iron control in chemical processing solutions. Chloride technology is being used on a limited scale in Europe and this technology si being advocated in North America. In Europe, because of disposal regulations, emphasis will be placed on the production of a useful iron product; in Canada, the objective would be to form an environmentally acceptable residue for disposal.

Activities in this areas will be outlined as follows

1. A.J. Monhemius (Imperial College) and Prof. Gaunand (Ecole des Mines) will continue to develop the solvent extraction process for iron, but will shift his emphasis to chloride solutions. This technique seems quite capable of yielding a marketable product, and there is commercial interest in this technology.

   Effort will be made to run its proces on a continuous basis to simulate plant practice. A second area of involvement will be characterization of chloride complex in concentrated iron solutions at high temperature using spectrophotometric methods. This work is of long-term importance to the iron control program, and would provide useful data in this area. Details of the proposal are contained in the submission of Dr. Monhemius.

2. Minemet/Air Liquide : Their are supporting Dr. Monhemius' work on hematite precipitation, but will emphasize FeO.OH precipitation from chloride solutions. Work will study the kinetic aspect and the use of high pressures of oxygen for high heat conservation and kinetic reasons. These companies will also undertake processes development work in this area, which emphasis on design and energy considerations. Details are given in the separate submission of F. Ricalens.

3. Canadian Participation : Bacon, Donaldson Associated of Vancouver is currently investigating iron control in chloride solutions under government contrat and will likely continue to work with interest in this field.

   CANMET has done some work on iron precipitation as hematite from chloride solutions and proposes to continue these activities with emphasizes on the following areas :

   Phase 1 : Identification of the conditions of temparature, concentration of Fe^{2+} and Fe^{3+} and pH which affect hematite precipitation from chloride solutions. The reaction will be followed in terms of extent of iron precipitation and on the

chemical and physical forms of the product. A careful mineralogical study will be made of the modification of FeOOH into Fe_2O_3

Phase 2 : The second phase will be done to ascertain the extent of impurity incorporation in the product as a function of the impurity levels in the precipitate. The general preferred precipitation conditions will be identified in phase I and the impurity level will be varied over the width ranges likely to be encountered in industry. Careful mineralogical work will be done to ascertain whether impurities are incorporated with hematite or are present as crystalline or amorphous phases. Impurities to be studied will include SO_4, Zn, Cu, Pb, As, Ag, etc.

Phase 3 : Current studies on goethite precipitation in sulphates media will be extended to chloride solutions with special emphasis on the content and volume of impurity incorporation. Several ions such as SO_4, Cl, Zn, Cu,; Pb, Ag, etc. will be studied. Efforts will be made to define the extent of impurity incorporation as a function of the precipitation conditions, and to determine whether the impurities are incorporated into the FeO.OH, or are adsorbed on the FeO.OH or are present as impurity phases.

Each phase of the CANMET work will likely require about a year to complete, and it is prepared to the work in the order listed.

IRON CONTROL IN COMPLEX ORE LEACH LIQUORS BY HYDROLYTIC STRIPPING

IMPERIAL COLLEGE

A.J. MONHEMIUS

Objectives

1) Production of pure hematite from chloride solutions containing iron, copper and other metals by the use of solvent extraction and hydrolytic stripping.

2) Measurement of thermodynamic data necessary to understand the equilibrium chemical behaviour of concentrated chloride solutions containing copper and iron.

PROGRAMME OF RESEARCH - ITEM 1 HEMATITE PRODUCTION

The work will continue and develop the results and experience obtained in the current EEC contract (MSM 094 UK). In particular emphasis will be placed on continuous miniplant experiments in order to overcome some of the problems of non-reproducibility experienced with batch precipitation experiments.

i) Assembly and commissioning of continuous autoclave miniplant system. The plant will consist of a small multistage, continuous stirred autoclave (2 l capacity), with a pressure pump for solution feed, a system for slurry discharge under pressure into a second holding autoclave and a pressure filter for solid-liquid separation. Most of this equipment is already available at Imperial College.
(Execution 1986).

ii) Use of this miniplant for precipitation of hematite from iron-loaded versatic acid solutions. Collection of data for the engineering design and economic evaluation of the process, e.g.residence time requirements, filtration characteristics, organic recovery methods, process control strategies, etc.

iii) Initially hematite precipitation will be carried out using batches of iron-Versatic solutions prepared from synthetic Fe/Cu chloride solutions, using extraction conditions established by the work of Dr. A. Gaunand at the Ecole des Mines in Paris.
(Execution 1978-88)

iv) At a later stage, Fe/Cu solutions obtained by leaching complex sulphide ores will be used in place of synthetic solutions in order to investigate impurity levels in the precipitated hematite. The physical and chemical characteristics of hematite produced by the organic route, together with estimated production costs, will be compared and contrasted with hematite produced by direct precipitation from aqueous chloride leach liquors. The acqueous precipiation route will be studied by CANMET

(Execution 1988-89)

v) A feasibility study will be carried out into the industrial utilisation of hematite produced from hydrometallurgical processes. Market sizes, physical and chemical specifications, price structures, etc. will be investigated for the various possible outlets for chemically-produced iron oxide, e.g. pigments, iron powder production, steelmaking via directly-reduced iron pellets or plasma processes, etc.

ITEM 2 - THERMODYNAMIC STUDY

A study will be carried out into metal speciation at elevated temperatures in concentrated chloride solutions containing iron and copper. This will involve the use of spectrophotometry to determine the stability constants of the various metal chlorocomplex species. A pressure spectrophotometer, designed and built at Imperial College, is available for this work.

These basic thermodynamic data are of fundamental importance for understanding the chemistry of many chloride processes, including those being developed by EEC contractors and CANMET. It is intended that this work will be carried out by a postgraduate student as a research project for a PhD thesis.
(Execution 1986-89)

COLLABORATIONS

i) The existing collaboration with Dr. A. Gaunand at the Ecole des Mines, Paris, will continue.

His work on the use of 3-phase extraction to transfer iron into Versatic acid is of primary importance in the application of hydrolytic stripping to complex ore treatment.

ii) The relative benefits and process economics of the hydrolytic stripping route will be compared and contrasted with the work at CANMET on the precipitation of hematite from aqueous chloride solutions and with the work of l'Air Liquide/Minemet on the precipitation of goethite.

iii) The thermodynamic information produced by the study outlined in Item 2 above will be of direct benefit to the EEC projects at Ecole des Mines, at l'Air Liquide and at Minemet and also to the various Canadian chloride leach processes under development at CANMET and elsewhere.

Contrôle du fer en hydrométallurgie

MINEMET RECHERCHE

F. RICALENS

But de la recherche

Les lixiviations en milieu chlorure de minerais complexes et de chalcopyrite mettent en solution des quantités notables de fer. Ces ions ferreux constituent un gêne dans la suite du procédé et doivent être purgés.

La purge doit être réalisée de manière que ni la formation du composé ferreux ni sa mise à la décharge ne soient coûteuses. L'idéal serait de trouver une utilisation à ces rejets de fer.

Un des points clés est l'entraînement dans le composé ferreux des éléments présents de la solution de chlorure. Des résultats substantiels ont déjà été obtenus par application de la technique de précipitation de goethite.

Le problème ci-dessus se pose de manière encore plus aigue lors de la valorisation des pyrites par un procédé analogue pour donner du soufre.

Le but de la recherche est de perfectionner le procédé "goethite" et de comparer les performances des divers autres procédés de purge de fer.

Programme de recherche

1ère phase (18 mois)

a) Etude des conditions de précipitation de la goethite

L'étude portera surtout sur la détermination des conditions de précipitation conduisant à un entraînement aussi faible que possible des métaux lourds d'une part et des ions chlorure d'autre part, et ce à des conditions économiquement acceptables (cette partie de l'étude devrait pouvoir être menée avec l'AIR LIQUIDE).

b) Etude des contraintes relatives aux diverses utilisations des différents oxydes et hydroxydes de fer

Des contacts devront être pris avec les gros utilisateurs d'oxydes de fer et avec leurs centres de recherche afin d'appréhender le plus complètement possible les contraintes et les moyens d'y remédier.

2ème phase (18 mois)

c) Etude comparative avec divers autres procédés (extraction liquide-liquide ou précipitation d'hémtatite in situ) (12 mois)

Ces études, qui devront être menées uniquement à l'échelle laboratoire, devraient permettre de déterminer les procédés à choisir suivant les conditions économiques et les buts à poursuivre. Ces études devront déboucher sur

d) une étude économique comparative (4 mois)
e) étude en continu (12 mois)

Cette étude optionnelle devra permettre de vérifier en continu (micropilote) l'utilisabilité du procédé choisi (conditions opératoires).

Si un accord de recherche a pu être conclu avec une société sidérurgique, la quantité de résidu formé devra permettre un essai d'utilisation dans les installations de recherche de ladite société.